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GESP-436

CORROSION TESTS OF LITHIUM FLUORIDE IN CONTACT WITH COLUMBIUM ALLOYS

TOPICAL REPORT II

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LITHIUM FLUORIDE BELLOWS CAPSULE TESTS

prepared by

R. W. Harrison
W H. Hendrixson

January 29, 1970

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-8523
J. A. Milko, Project Manager
Materials Section

NUCLEAR SYSTEMS PROGRAMS
SPACE SYSTEMS
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

NOTICE

GESP-436

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LITHIUM FLUORIDE BELLOWS CAPSULE TESTS

I. INTRODUCTION

The selection of the thermal energy storage material for an earth orbiting, solar dynamic power conversion system must be based on its thermal properties as related to the thermodynamic cycle of the system. The desirable properties would include a melting point slightly higher than the temperature capabilities of the rotating equipment, a high heat of fusion and heat capacity combined with a low density to minimize weight. A reasonable high thermal conductivity is desired for both the liquid and solid state to transfer heat efficiently from the heat adsorbing surface of the cavity receiver to the working fluid of the power conversion system. In addition the following properties are desirable to design a thermal storage system of minimum weight: a low volumetric phase change, a low vapor pressure at the working temperature, high-temperature chemical stability, and compatibility with the containment material.

An early study[1] on a Brayton Cycle system designed to operate at turbine inlet temperatures of approximately 1200°F used lithium hydride (melting temperature, 1265°F) as the heat storage material. Use of lithium fluoride as the heat storage material permits an increase in the turbine inlet temperature to approximately 1500°F due to the higher melting temperature, (1560°F) of this salt.[2] In addition the use of lithium fluoride, rather than the hydride, avoids the difficult problem of containing dissociated hydrogen which is encountered in lithium hydride systems at elevated temperatures.

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(1) "Sunflower Boiler/Heat Storage Topical Report," TRW, Inc., ER-4869, April 1963.

(2) "Brayton Cycle Cavity Receiver Design Study," NASA CR-54752, November 2, 1965.

Lithium fluoride has excellent thermal properties in the 1550°F to 1900°F temperature range. It also has a low vapor pressure and does not dissociate in the temperature range of interest. The significant properties of LiF for this application are presented in Table I.

A variety of conventional iron, nickel and cobalt-base alloys have been evaluated as potential containment alloys for lithium fluoride heat storage systems. The results of experiments,[3,4] which were conducted for time periods of approximately 10,000 hours on these alloys, indicated that of the various compositions tested, the cobalt-base material Haynes Alloy No. 25 was best suited for use as a material of construction in heat storage systems on the basis of corrosion resistance and metallurgical stability. However this study further concluded, that high-temperature creep strength testing would be required in the temperature range of interest to confirm the value of Haynes Alloy No. 25 in a specific application. A brief comparison of the strength properties of this superalloy and the proposed columbium-base alloys indicated that columbium-base alloys had considerable potential for use in high temperature lithium fluoride heat storage systems.

The purpose of the proposed program, then, was to determine the compatibility of several promising columbium-base alloys with lithium fluoride under the cyclic thermal conditions which simulate the sun-shade cycle of a heat receiver, and to evaluate a design concept for containment of lithium fluoride in a manner to accommodate the 29% expansion on melting. The compatibility tests indicated the columbium-base alloys Cb-1Zr, FS-85, and SCb-291 were corrosion resistant to lithium fluoride. The results of this study are described in a previous report.[5]

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[3] Schulze, R. C., "The Corrosion of Superalloys by Lithium Fluoride in a Cyclic High Temperature Environment," NASA CR-54781, June 21, 1965.

[4] "Brayton Cycle Cavity Receiver Design Study," NASA CR-54752, November 22, 1965.

[5] Harrison, R. W. and Hendrixson, W. H., "The Compatibility of Columbium Base Alloys with Lithium Fluoride, Topical Report No. 1, NASA Contract NAS 3-8523, GESP-261, September 1969.

TABLE I.

PHYSICAL PROPERTIES OF LITHIUM FLUORIDE

Density	(solid: 25°C, 77°F) 2.63905 ± 0.0001 gm/cm^3
	(liquid: 870°C, 1598°F) 1.789 gm/cm^3
	(liquid: 887°C, 1629°F - 1058°C, 1936°F)
	$d_4^t = 2.201 - 0.000474t$ gm/cm^3
Melting Point	848°C ± 1° (1558°F ± 2°)
	(ΔV on melting = +29.4%
Boiling Point	1681°C (3058°F)
Heat Capacity	C_p (solid: 25°C, 77°F) = 10.015 cal/degree/mole
	C_p (liquid: 848°C, 1568°F - 896°C, 1645°F) = 15.51 cal/degree/mole
Heat of Fusion	$\Delta H_{848°C,\ 1568°F}$ = 6.470 kcal/mole

Vapor Pressure of Fused Salt	Temp. °C	Temp. °F	mm of Hg
	1047	1917	1
	1211	2212	10
	1333	2431	40

Since the initiation of this program the Solar Brayton Cycle Heat Receiver has been designed, fabricated, and was recently shipped from GE-NSP to NASA. The heat receiver, shown during a phase of fabrication in Figure 1, consists of forty eight tubes which are connected between the inlet and outlet gas headers. Each of these tubes is fabricated from a bellows which forms the outside of the tube, or heat absorbing surface, and an inner tube through which inert gas flows. Lithium fluoride is contained in the annular space.

Three Cb-1Zr capsules of similar configuration to the heat receiver tubes were tested under the cyclic thermal conditions which simulate the sun-shade cycle of a heat receiver in a 300 nautical mile orbit. The results of these tests, which are presented in this report, were used in establishing the design and procedures used in the construction of the Solar Brayton Cycle Heat Receiver.

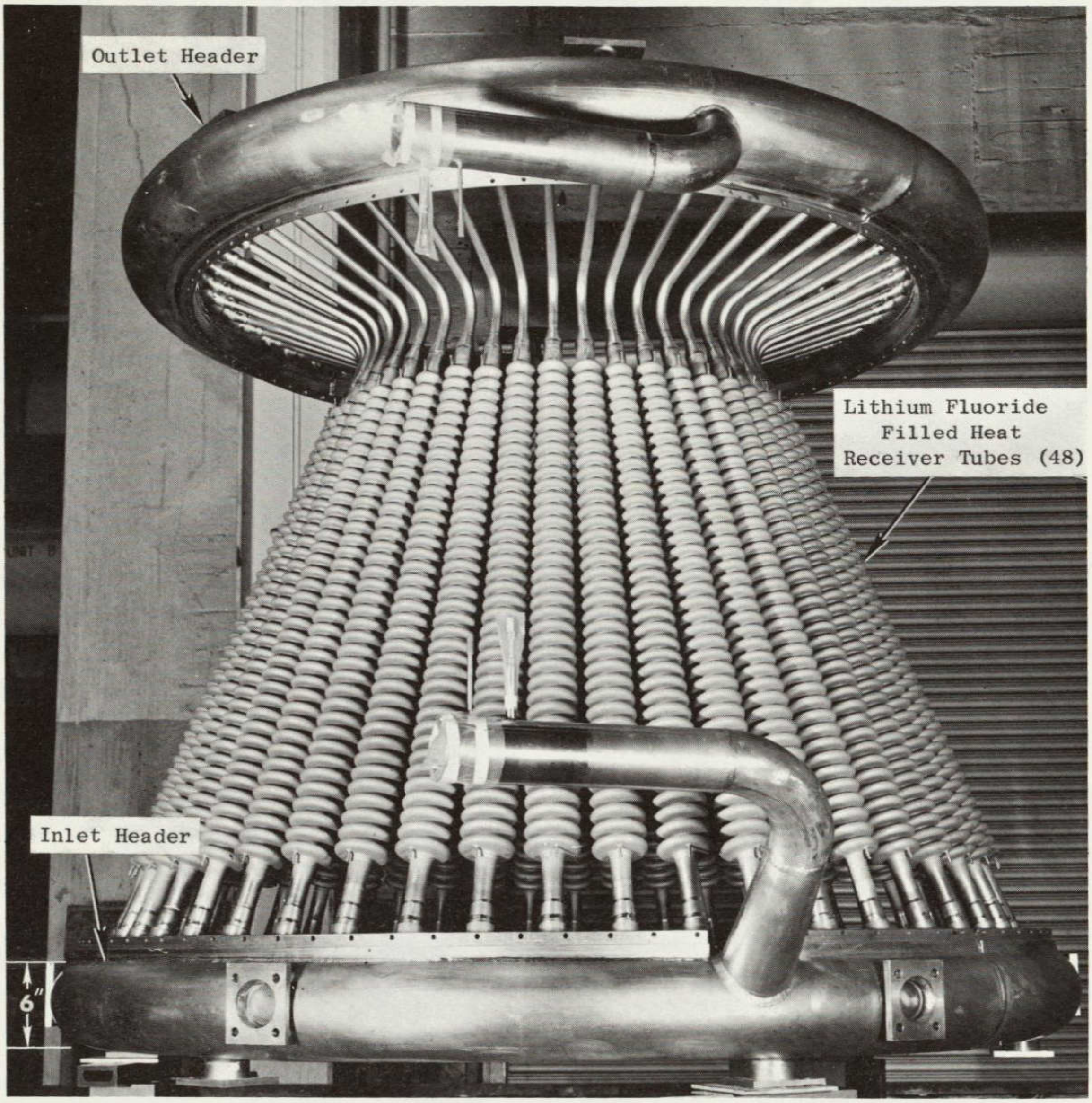

Figure 1. Cb-1Zr Solar Brayton Cycle Heat Receiver During Fabrication. (P69-11-12D)

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II. EXPERIMENTAL PROCEDURE

A. CAPSULE FABRICATION

The bellows assemblies of each capsule were fabricated by electron beam welding die formed segments, each segment representing one-half of a convolution. To maintain concentricity, segment pairs were circumferentially welded on the ID and placed on a mandrel before the OD circumferential welds were made. Inner tubes of the proper size were used to maintain a nominal 0.010 inch annulus between the ID of the bellows assembly. Prior to welding all Cb-1Zr parts were pickled to NSP Specification 03-0021-00-A "Cleaning and Handling of Components for Alkali Metal Service." A cap was welded on the top end of the inner tube which was then inserted inside the bellows assembly. The two were then welded together at the bottom and the filling chamber was welded on to the top of the bellows.

All welding was performed to NSP Specification 03-0025-00-A "Welding of Columbium, Tantalum and Their Alloys by the Inert Gas Tungsten Arc Process" the capsule was determined to be free of leaks when tested according to NSP Specification 03-0013-00-B "Mass Spectrometric Leak Detection Using Helium."

The convolution geometry of all the capsules were similar to the largest convolutions on the tapered heat receiver tubes and measured 3.25-inch diameter.

1. Lithium Fluoride Bellows Capsule I

The first capsule, shown in Figure 2, was fabricated prior to the establishment of the heat receiver tube lithium fluoride filling facility at ORNL. As a result, this capsule was filled at GE-NSP and was fabricated with a filling chamber attached.

2. Lithium Fluoride Bellows Capsules II and III

Bellows Capsule II and III shown in Figures 3 and 4 more closely resemble the heat receiver tube geometry since they were fabricated and filled with lithium fluoride using techniques developed for the heat

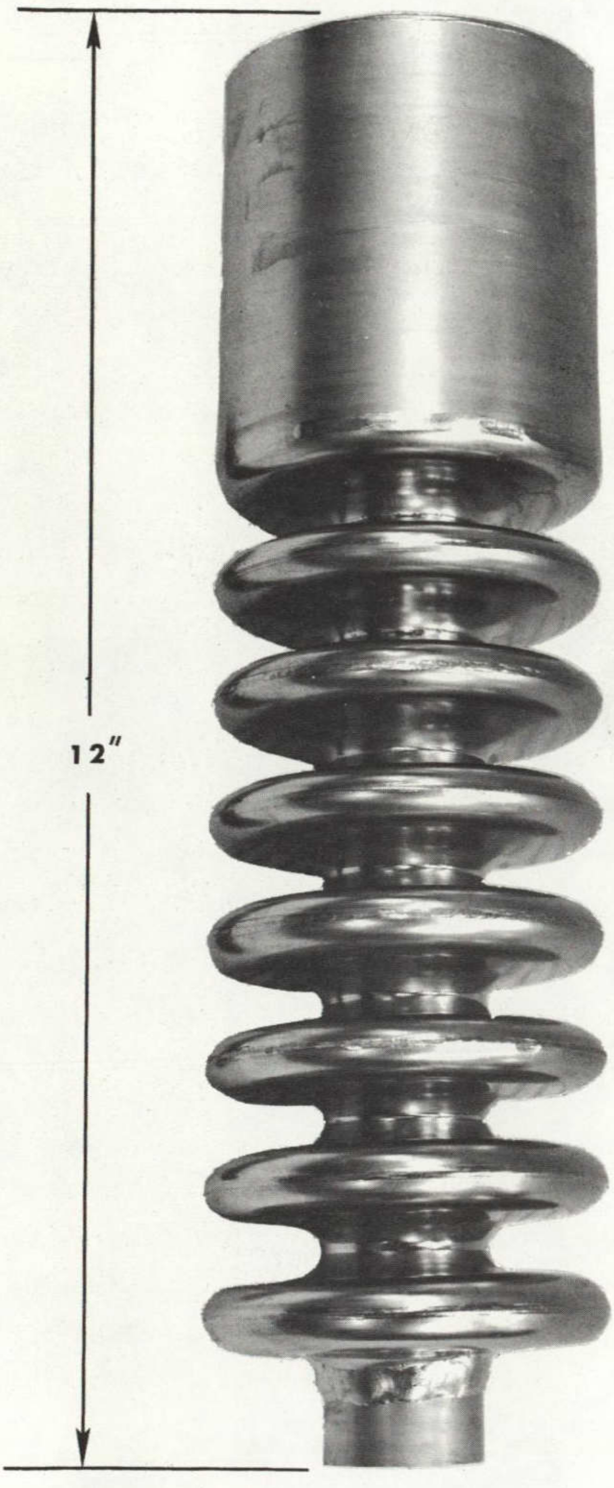


Figure 2. Cb-1Zr Bellows Capsule I Simulating a Lithium Fluoride Brayton Cycle Heat Receiver.

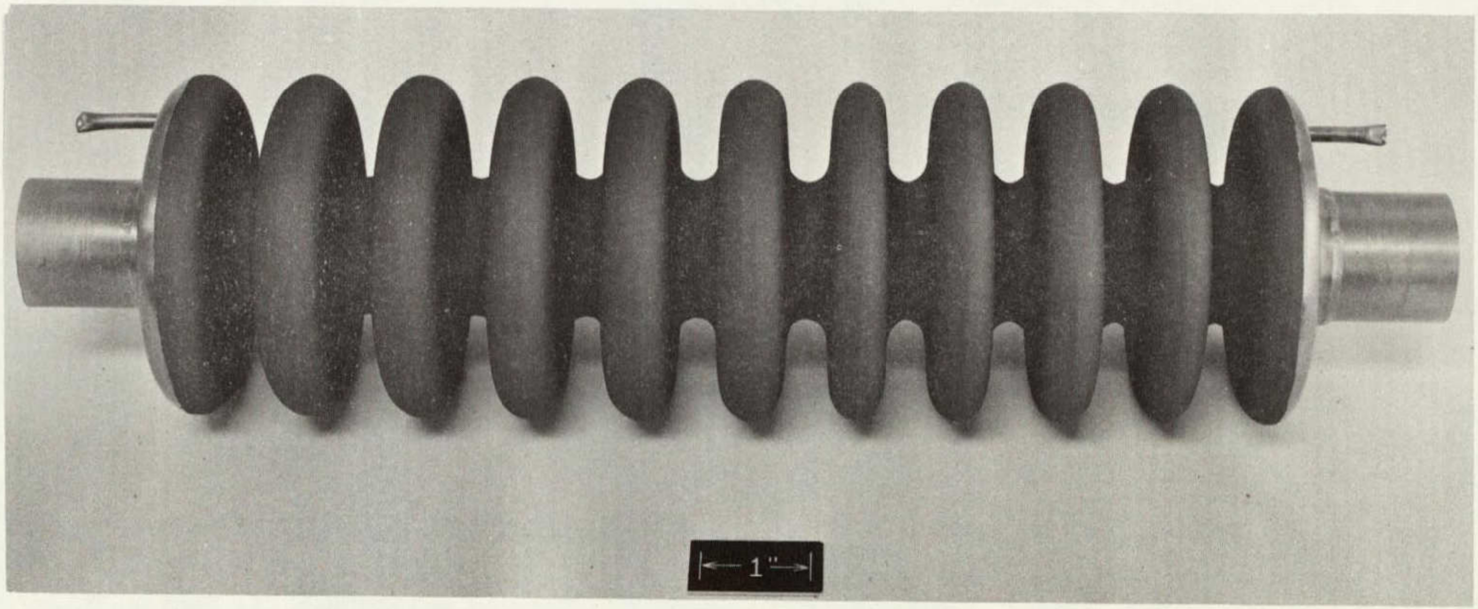
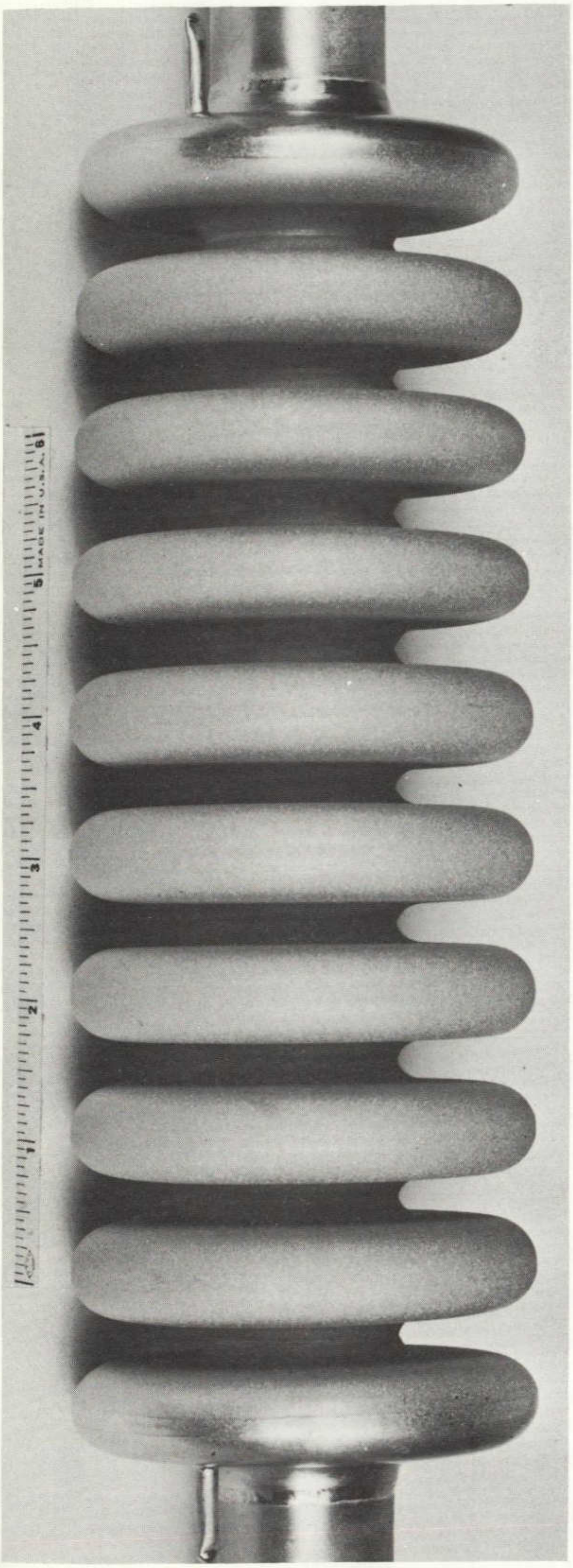
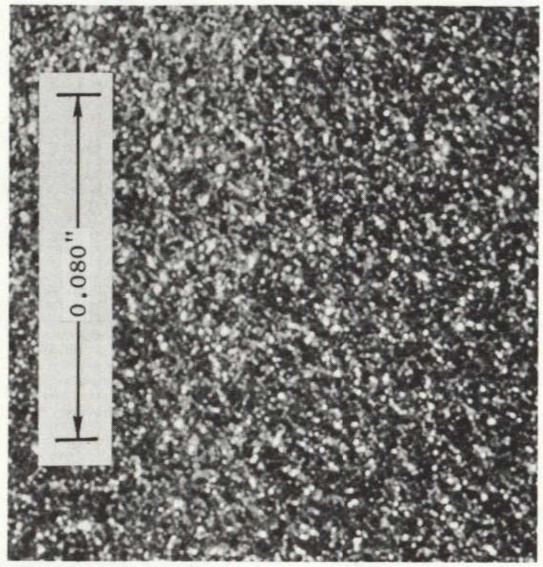


Figure 3. Iron Titanate Coated Bellows Capsule II. (C67091940)

E1288-2



(P68-12-9D)



Alumina Grit Blasted Surface.
(P68-12-9B)

Figure 4. Lithium Fluoride Bellows Capsule III.

receiver tubes. The capsules were fabricated at the NASA Lewis Research Center, filled with lithium fluoride at ORNL under separate NASA contract, and delivered to GE-NSP for testing. Bellows Capsule II was coated with iron titanate and Bellows Capsule III was grit blasted to increase the absorptivity of the surfaces.

B. CAPSULE FILLING

1. Bellows Capsule I

The bellows capsule was filled with lithium fluoride at NSP Evendale. The bellows capsule filling facility is shown in the welding chamber in Figure 5. The capsule was positioned in the split tantalum heating elements by means of the Cb-1Zr cooling tube and instrumented with thermocouples on the filling chamber top and the bottom convolution as shown in Figure 6. It was necessary to use a Cb-1Zr cooling tube during the filling operation rather than a stainless steel cooling tube of the type to be used in the test facility since the cooling tube would reach the capsule temperature (1900°F) during filling. The vapor pressure of stainless steel at this temperature in vacuum would have resulted in contamination of the ID of the Cb-1Zr bellows capsule during this operation. Similarly helium was used for cooling to prevent oxidation of the inside surfaces of the Cb-1Zr cooling tube. The cooling tube was required in the filling operation for the same reasons as required in the test facility, namely, to produce a desirable lithium fluoride freezing pattern.

After bellows capsule and transfer container were placed in the welding chamber it was evacuated to 5.5×10^{-5} torr and backfilled with pure helium. Previously the lithium fluoride crystals for filling the bellows capsule had been weighed into stainless steel beakers, sealed and degassed in the transfer container.

The cube shaped lithium fluoride crystals measuring 5-15 mm on a side were carefully transferred from the beakers to the capsule filling chamber with a tweezers. After the calculated weight of 670 grams of lithium fluoride had been transferred into the capsule filling chamber, the lid was put in place, and the welding chamber was evacuated to

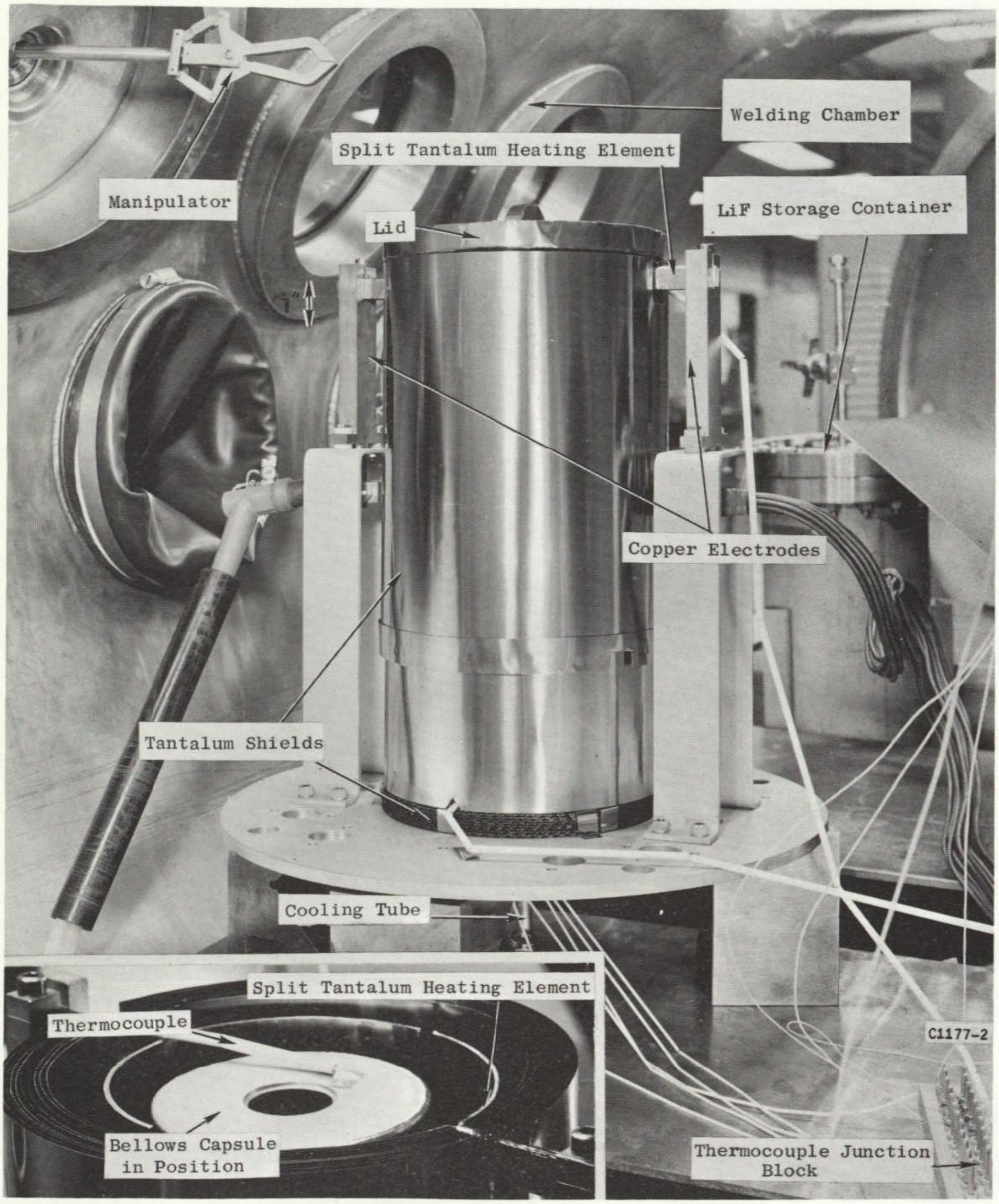


Figure 5. Bellows Capsule Lithium Fluoride Filling Facility Assembled in the Welding Chamber. Inset Photograph at Lower Left Shows the Top of the Assembly with the Insulating Lid Removed. (C67011370 & C67011351)

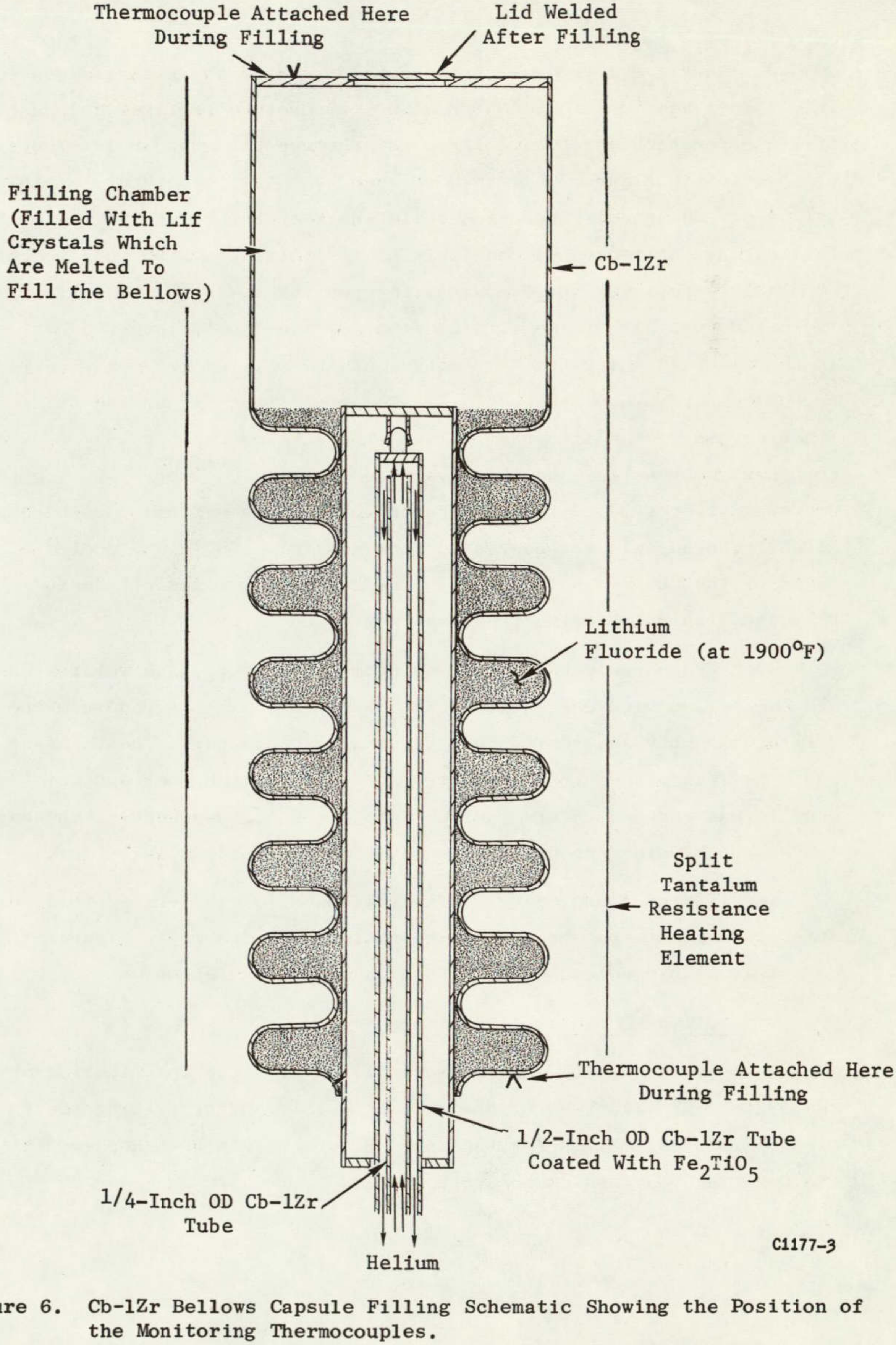


Figure 6. Cb-1Zr Bellows Capsule Filling Schematic Showing the Position of the Monitoring Thermocouples.

5×10^{-5} torr. Heating was initiated slowly with the pressure continuing to decrease to 3×10^{-5} torr. The temperature profiles during the filling operation as obtained from the thermocouples shown in Figure 6, are plotted in Figure 7. It can be seen that the temperature at the bottom of the capsule increased while the temperature at the top of the filling chamber decreased once melting was initiated with both reaching the same temperature three hours after heating was initiated. This indicated that all of the lithium fluoride had flowed into the bellows at the point at which the temperature of the filling chamber rose rapidly to the temperature of the bellows. To assure that all of the lithium fluoride had flowed into the bellows, the welding chamber was pressurized to 5 psia with helium and then evacuated to 3×10^{-5} torr. During the pressurization some cooling of the capsule by the helium atmosphere was noted. The capsule was heated to approximately 1900°F and cooled by means of the Cb-1Zr cooling tube. The heater power was off during the time the lithium fluoride freezing took place.

When the capsule had cooled to room temperature, the welding chamber was backfilled with pure helium and the capsule was removed from the filling assembly and sealed in the transfer container. The transfer container was placed in the electron beam welding chamber and the capsule was removed under a helium atmosphere. Subsequently, the capsule was sealed by electron beam welding in a vacuum of 1×10^{-5} torr.

The lithium fluoride filled bellows capsule was x-rayed to determine the freezing pattern obtained. The radiographs, shown in Figure 8, indicate that a void volume was obtained in each convolution.

2. Bellows Capsules II and III

Bellows Capsule II and III were filled with lithium fluoride at a temperature of 1775°F using the process developed for filling the full size bellows assemblies for the Solar Brayton Cycle Heat Receiver at the Oak Ridge National Laboratory (ORNL).

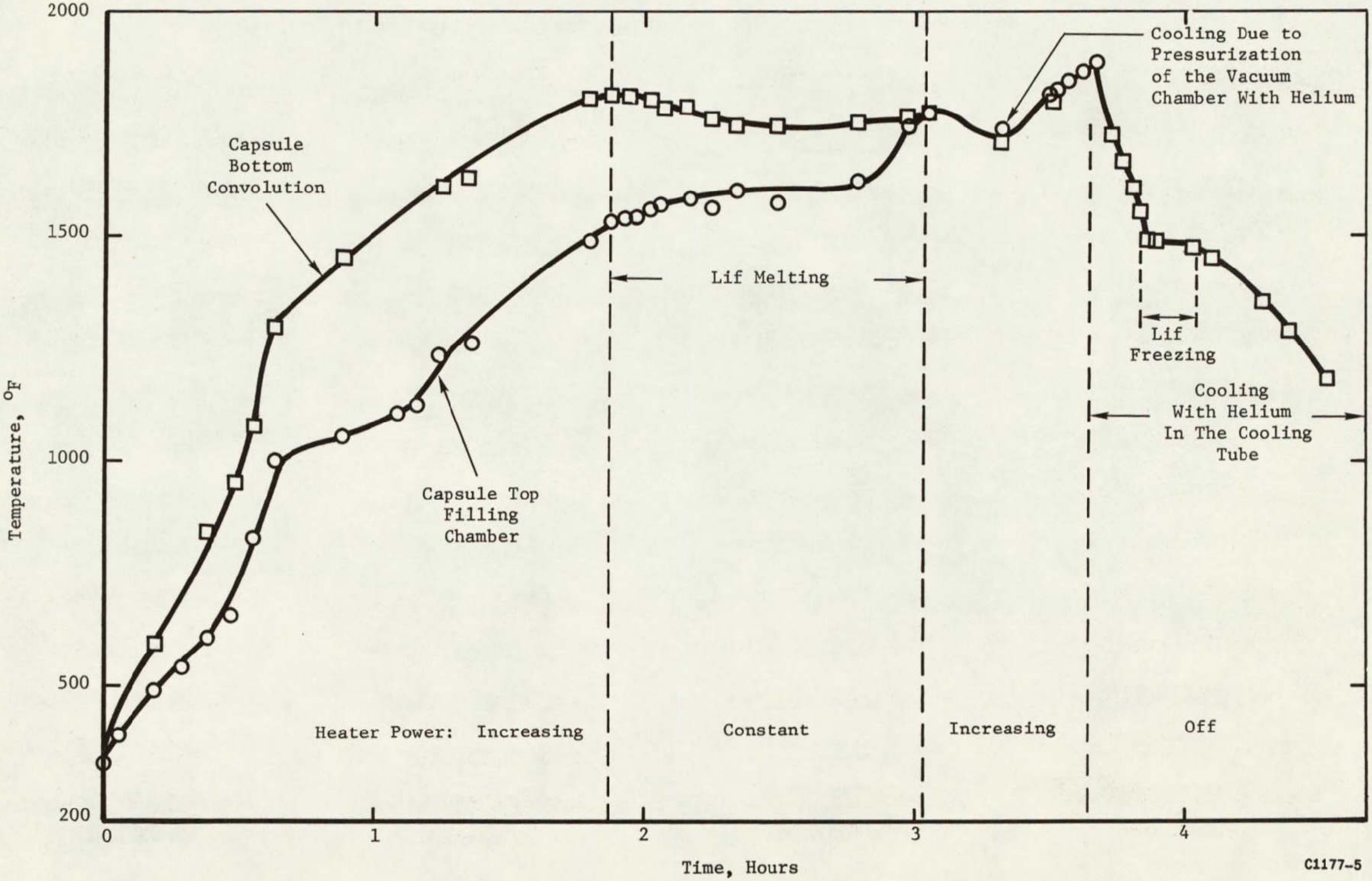


Figure 7. Temperature Profile of Bellows Capsule I During the Lithium Fluoride Filling Operation.

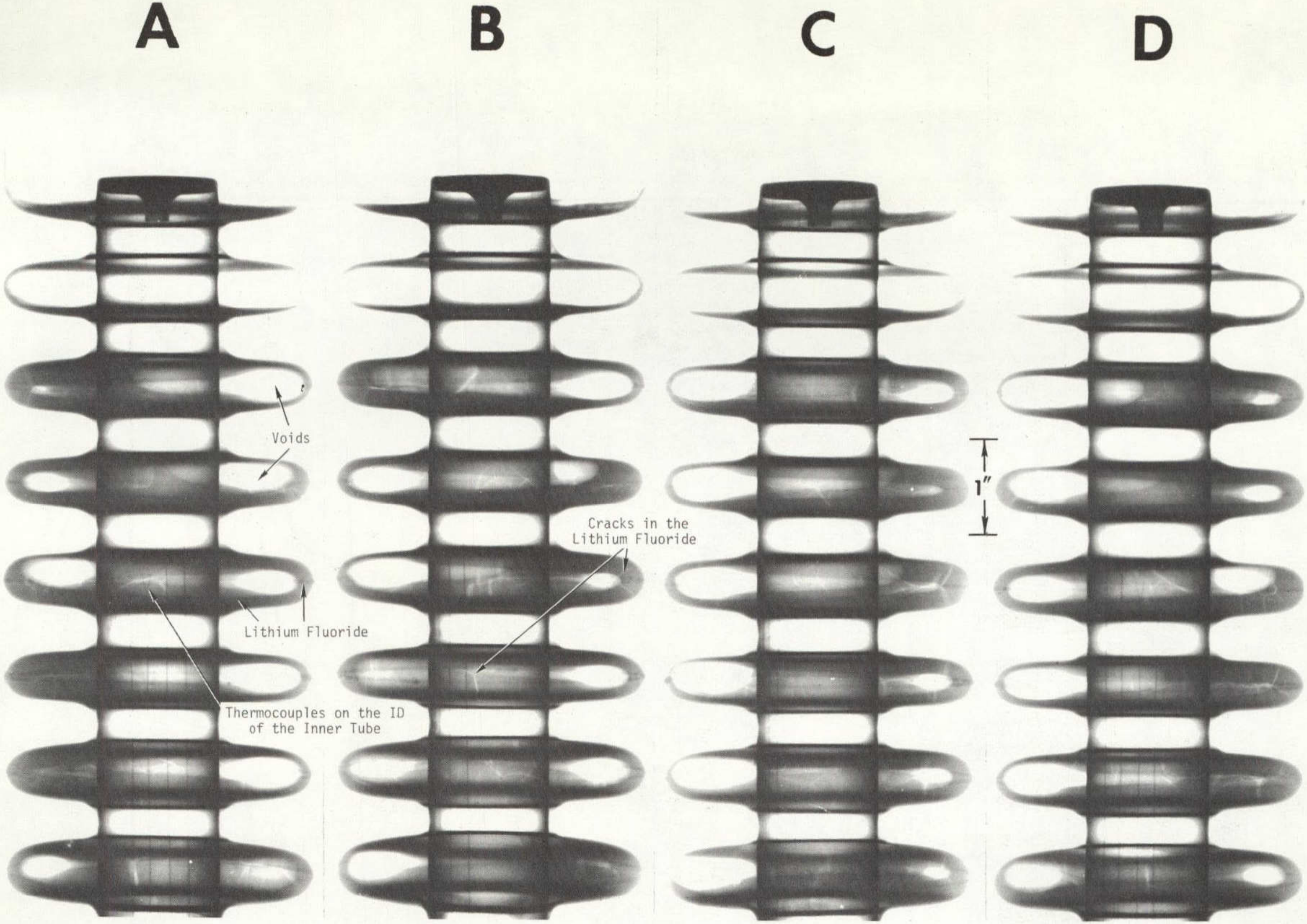


Figure 8. Radiographs of the Lithium Fluoride Filled Bellows Capsule I. AS865

Radiograph B Taken 45° from A.
Radiograph C Taken 90° from A.
Radiograph D Taken 135° from A.

C. CAPSULE TEST FACILITY

The lithium fluoride bellows capsule tests were conducted in an 18-inch-diameter Varian ultrahigh vacuum chamber. The complete bellows capsule test facility is shown in Figure 9.

1. Temperature Control System

The power for the tantalum heater was provided by means of a 10 kva step down transformer which reduced the line voltage from 220 volts to 12 volts. The line voltage which regulates the temperature was controlled by the temperature cycle control system, shown in Figure 10 which consisted of a cam-operated temperature programmer driving a silicon-controlled rectifier power regulator. The cam, shown in the temperature programmer in Figure 11 made one revolution every 96 minutes and was cut to provide two power settings. During the melting and heating cycle, a power value was applied, capable of heating the capsule to desired maximum temperature in one hour. This was accomplished by having the cam-actuated control set point well above the temperature indicator during the heating cycle, thereby, the programmer was constantly calling for maximum power. The maximum power level was in turn set by the current limited adjustment in the silicon-controlled rectifier power controller. After 60 minutes the cam lowered the set point to well below the indicated millivolt out put of the control thermocouple and shut the power off or reduced it to a minimal power which could be set with the minimum power adjustment in the silicon-controlled rectifier power controller. The low point of the cycle was selected to assure that all the lithium fluoride in the capsule had frozen before the power came on for the next cycle.

Power to the capsule heating element was delivered by measuring the voltage with a standard voltmeter (± 3 percent) and measuring the amperage with a Weston current transformer and an ammeter (± 0.5 percent) as shown in Figure 12.

2. Thermocouple Instrumentation

Each bellows capsule was instrumented with W-3Re/W-25Re thermocouples. All the thermocouples were attached according to NSP

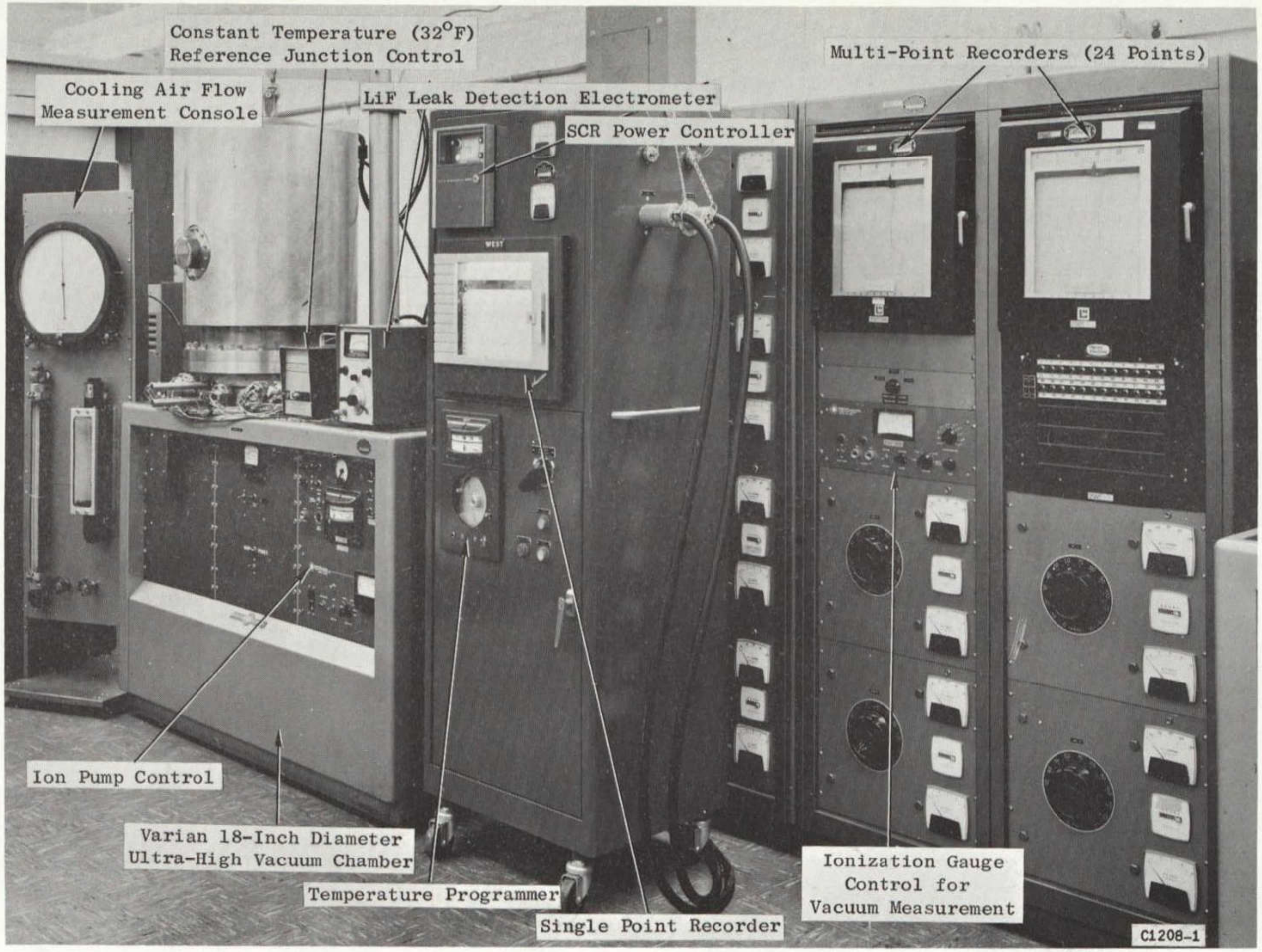


Figure 9. The Lithium Fluoride Bellows Capsule Test Facility. (C67031423)

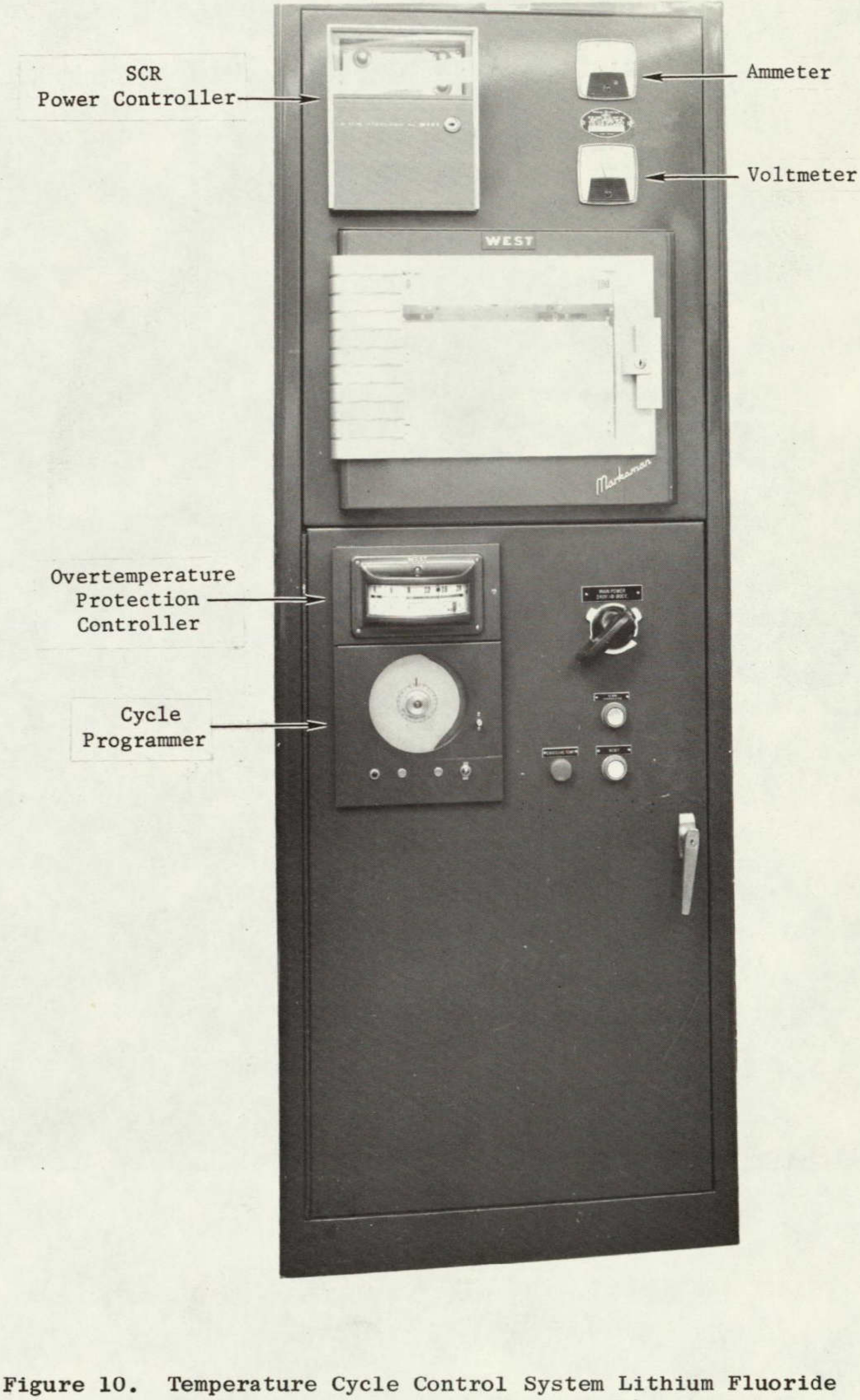


Figure 10. Temperature Cycle Control System Lithium Fluoride Bellows Capsule Tests. (C68022940)

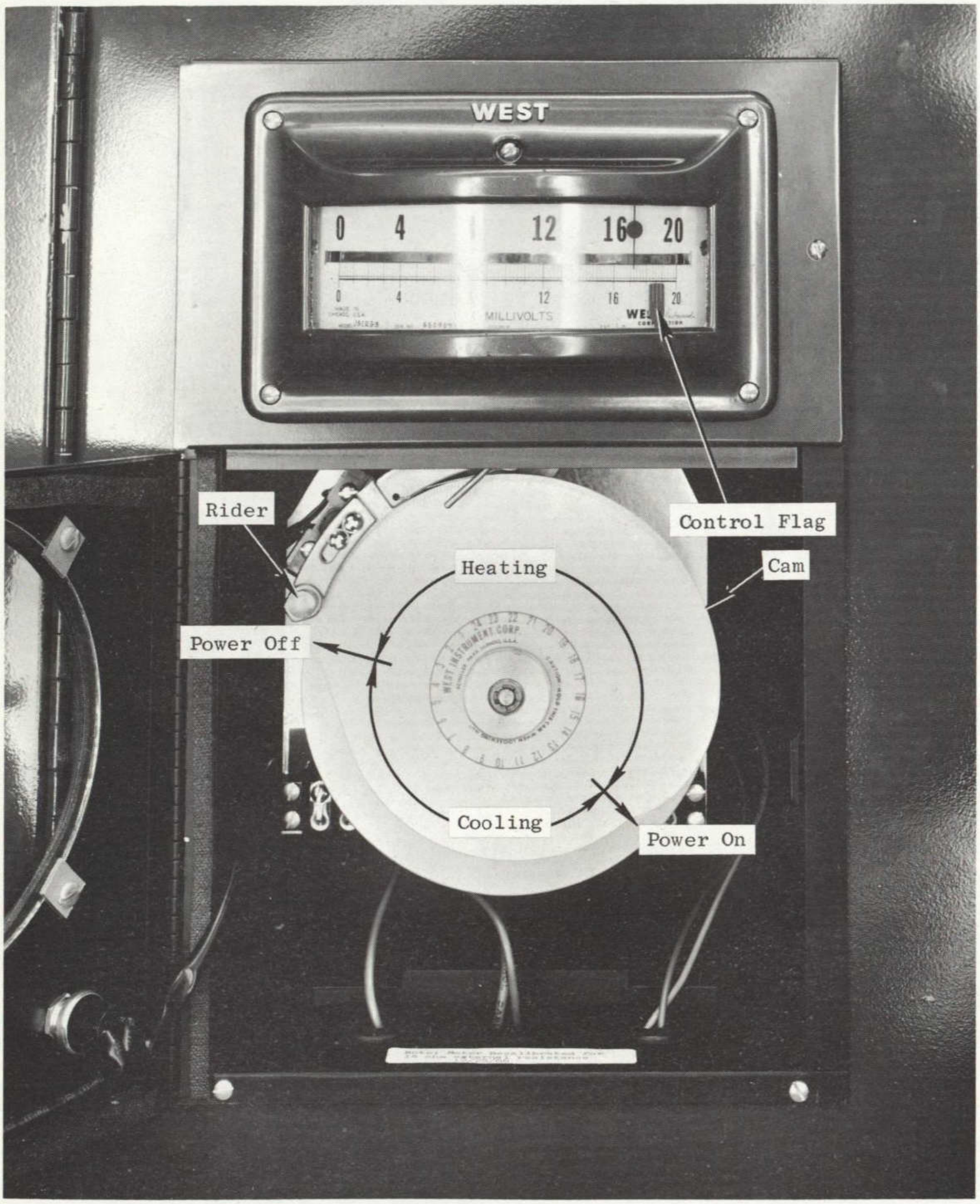


Figure 11. Cam Controlled Temperature Programmer - Lithium Fluoride Bellows Capsule Tests. (C67031427)

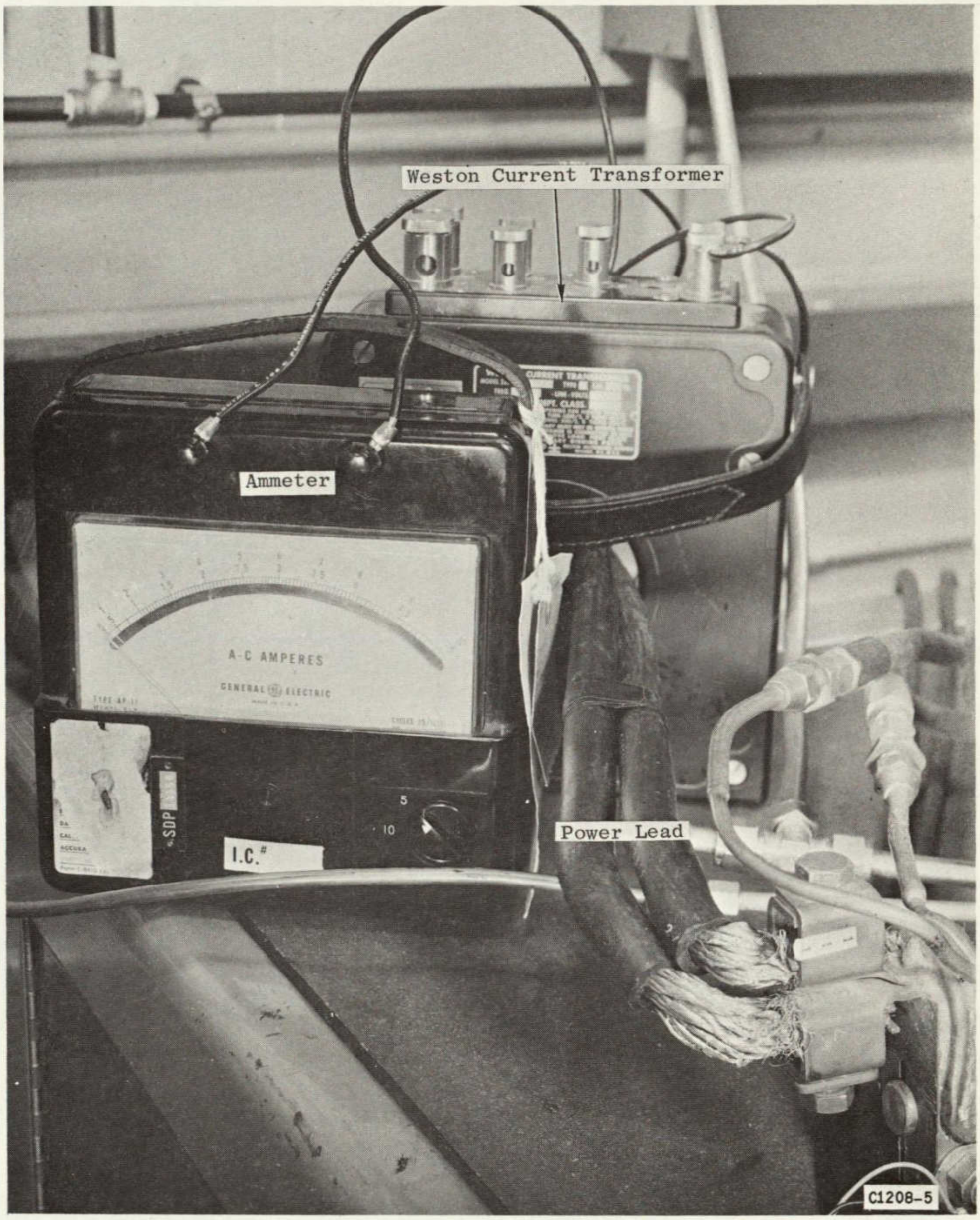


Figure 12. Current Measuring Instruments Employed in the Lithium Fluoride Bellows Capsule Tests. (C67031428)

specification 03-0019-00-A, "Thermocouple Installation Refractory Loop Systems in High Vacuum Environment." Instrumentation of Capsules I, II, and III was completed are shown in Figures 13, 14, 15, respectively. The iron titanate coating on the surface of bellows capsule II was removed in the designated thermocouple locations by alumina grit blasting small spots as shown in Figure 16. Separate spots were employed for attachment of each leg of the W-3Re/W-25Re thermocouples to minimize the uncoated wall area associated with the wall junctions and thereby improve the accuracy of the temperature measurement.

By incorporating a thermoelectrically cooled constant temperature (32°F) reference junction in the thermocouple circuitry, as shown in Figure 17, corrected temperatures were plotted on the recorder. Temperatures were periodically checked with a potentiometer by means of the thermocouple switches, shown in Figure 18 which switch the thermocouple input from the recorder into the potentiometer.

3. Lithium Fluoride Leak Detector

Lithium fluoride leak detectors were used on each of the bellows capsule tests. The operation of such a surface ionization detector depends on the conversions of an atom of low ionization potential into a positive ion on a surface with a high work function. The detector shown in Figure 19 employs a rhenium filament and a tantalum collector. Lithium fluoride molecules impinging on the heated rhenium filament will be dissociated to atoms and the lithium atoms subsequently will be ionized. The ionized lithium atoms will be drawn to the tantalum collector which is electrically negative. The electrical signal produced by these ions can be detected with an electrometer, as shown in the wiring schematic, Figure 20.

4. Cooling Air System

In the Solar Brayton Cycle Heat Receiver, the recirculating inert gas flows through the inner tube of the heat receiver tubes. A recirculating inert gas system for capsule testing over periods of 5000 hours was beyond the scope of this program. Instead, an iron titanate coated stainless steel cooling tube was used, and air was used as the cooling

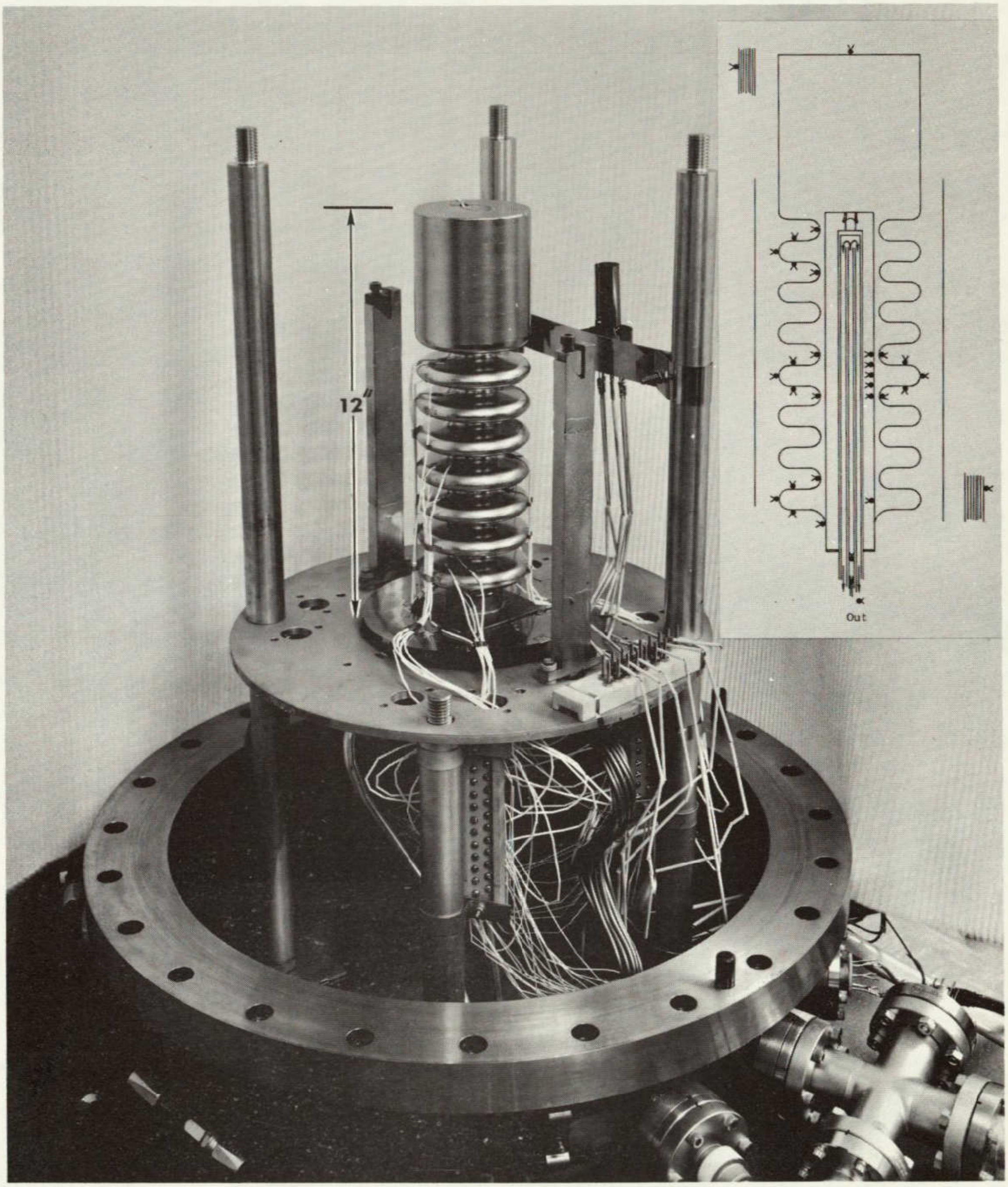


Figure 13. Thermocouple Installation Lithium Fluoride Bellows Capsule I. (C67021522)

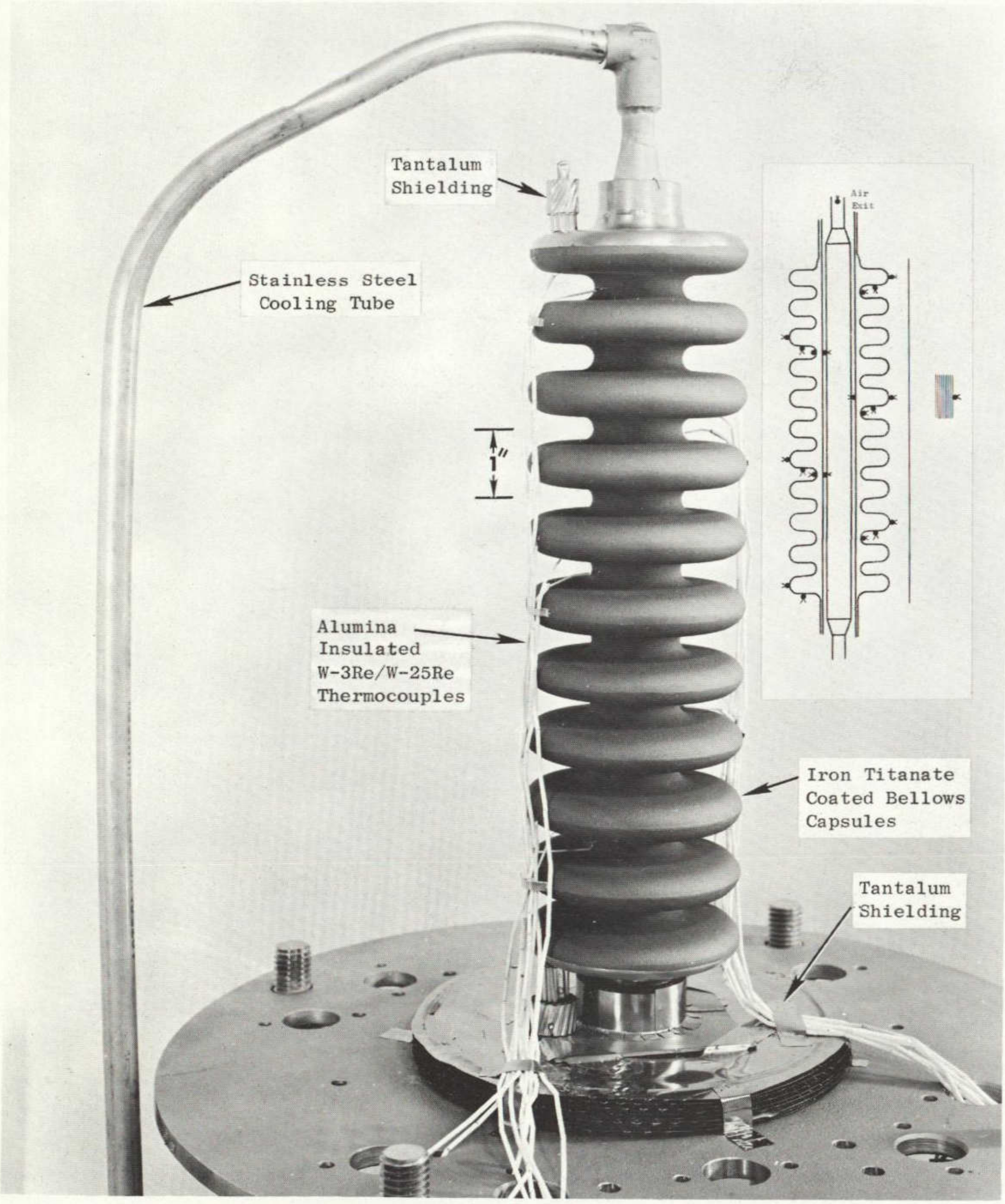


Figure 14. Thermocouple Installation Lithium Fluoride Bellows Capsule II. (C67102622)

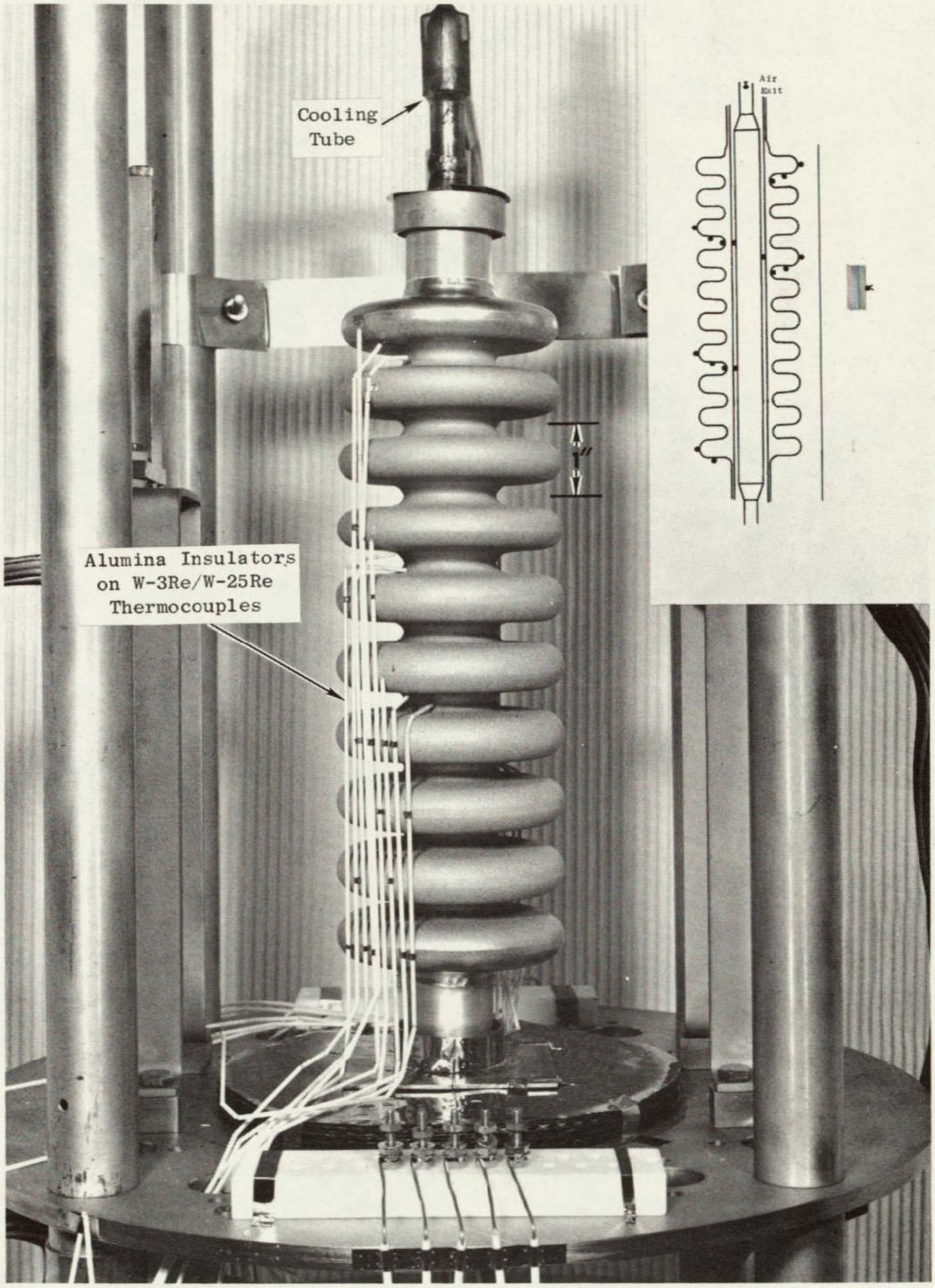


Figure 15. Thermocouple Installation Lithium Fluoride Bellows Capsule III. (P69-1-27E)

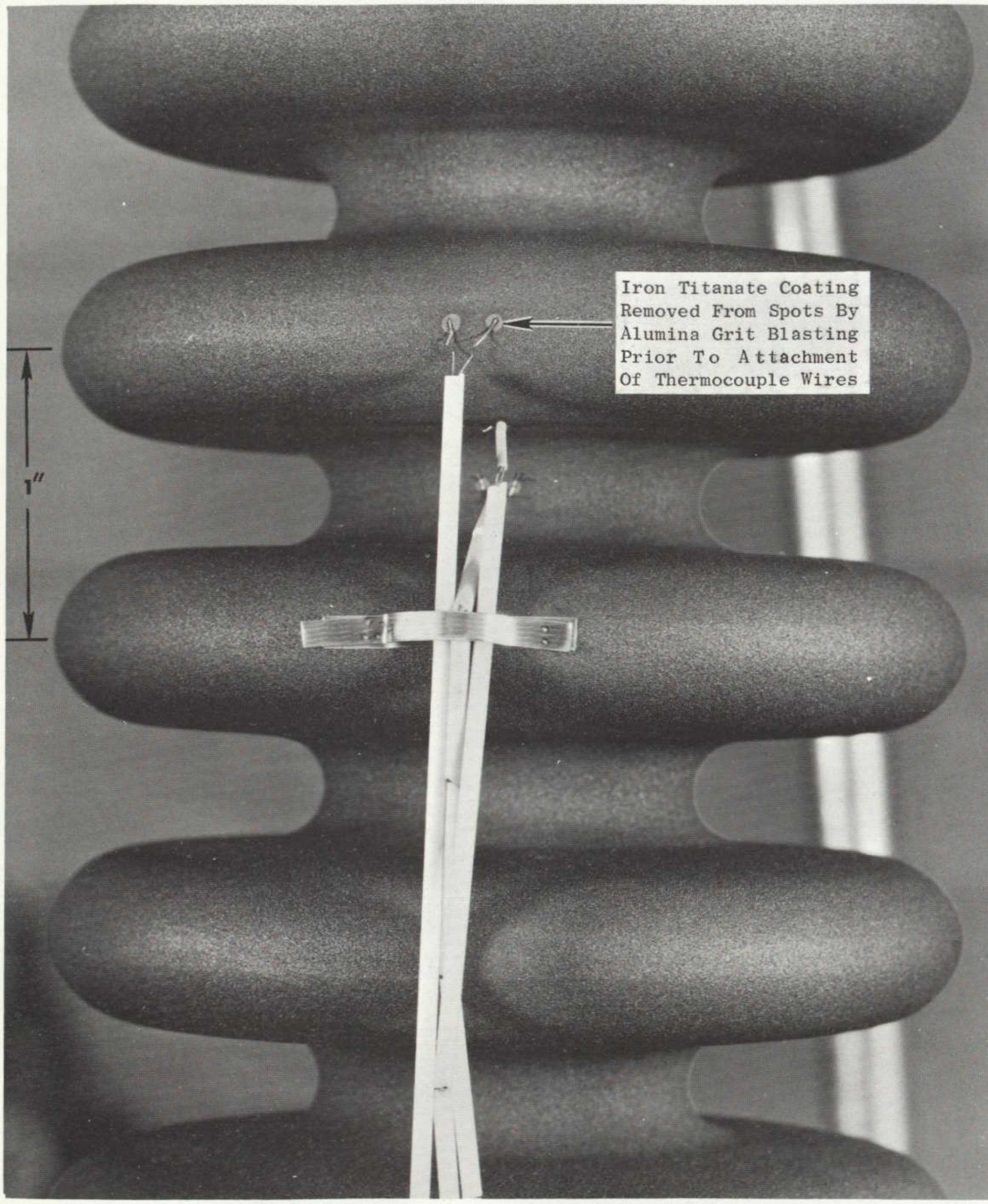


Figure 16. Thermocouple Installation Lithium Fluoride Bellows Capsule II. (C67102624)

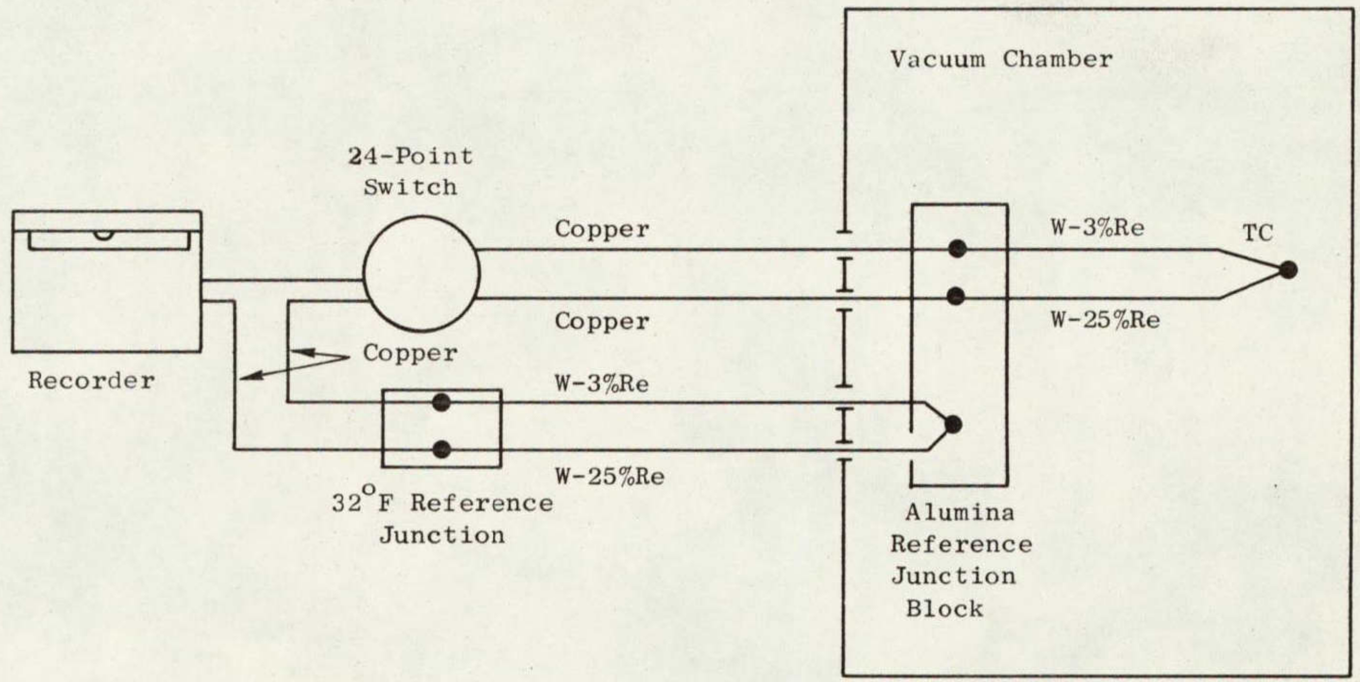


Figure 17. Thermocouple Circuitry Employed in the Lithium Fluoride Bellows Capsule Tests.

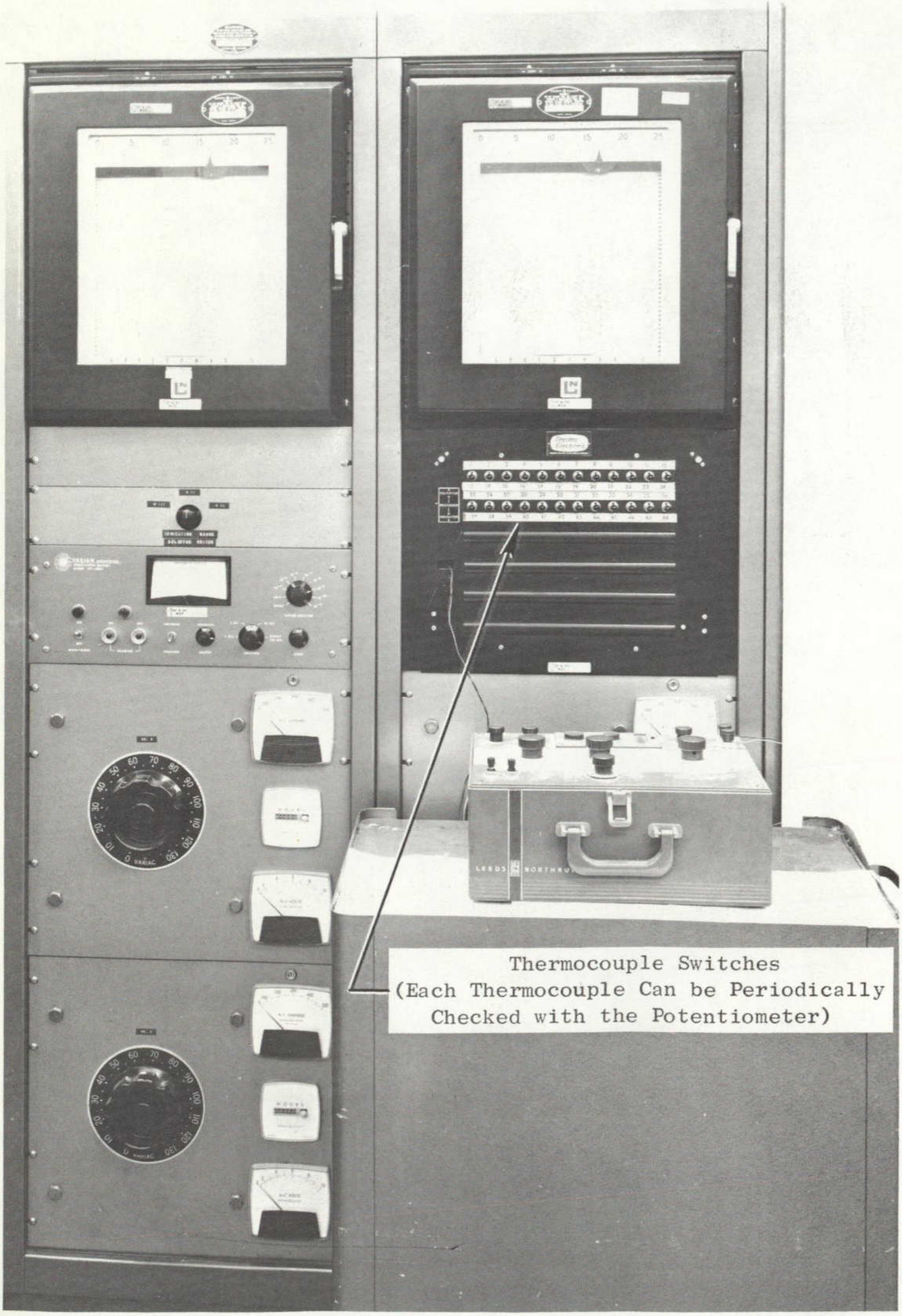
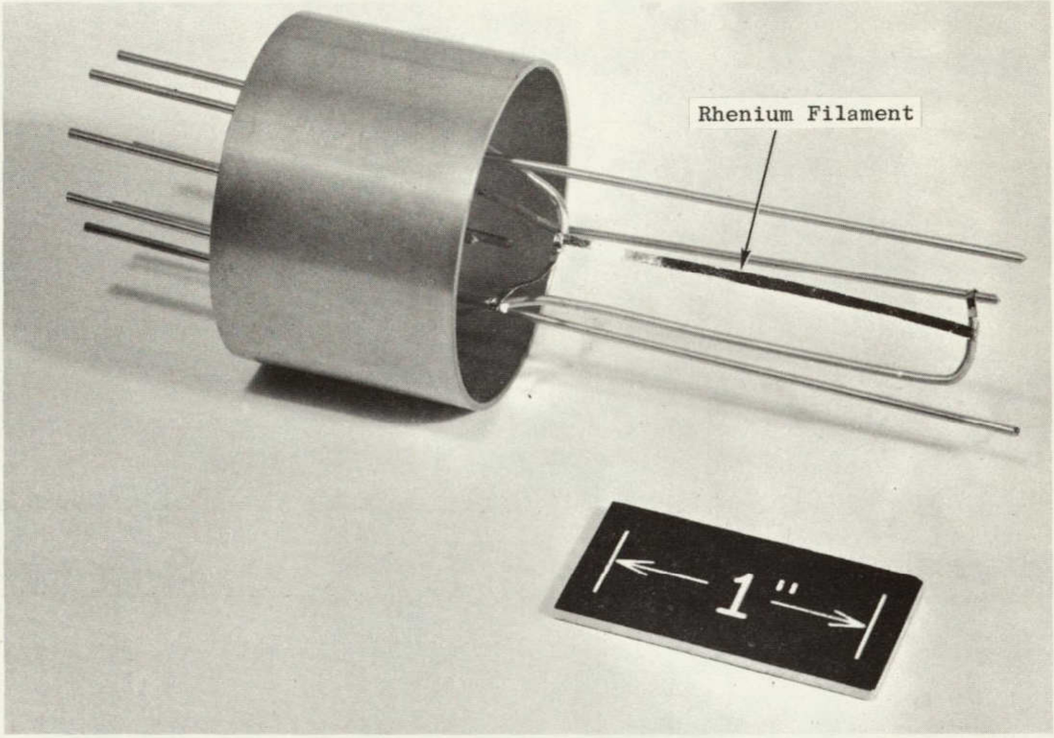
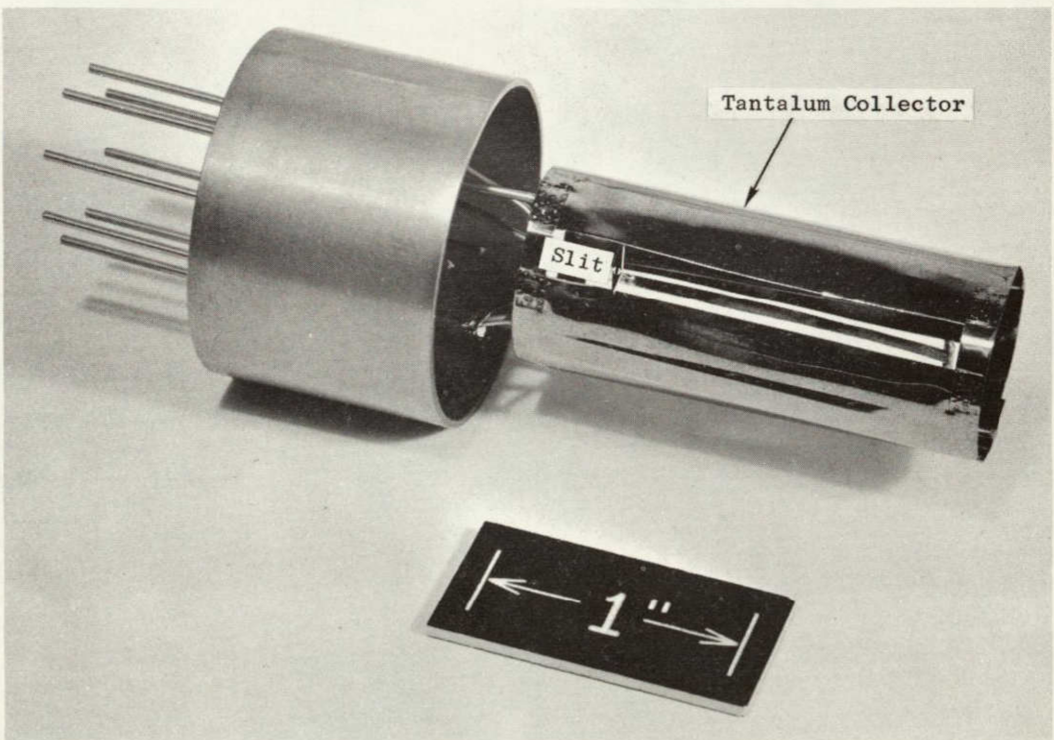


Figure 18. Temperature Measuring and Recording Instrumentation - Lithium Fluoride Bellows Capsule Test. (C67031432)



Before Attachment of the Tantalum Collector



Tantalum Collector In Place

Figure 19. Lithium Fluoride Leak Detector Filament and Collector Assembly. (C6122258 & C66122259)

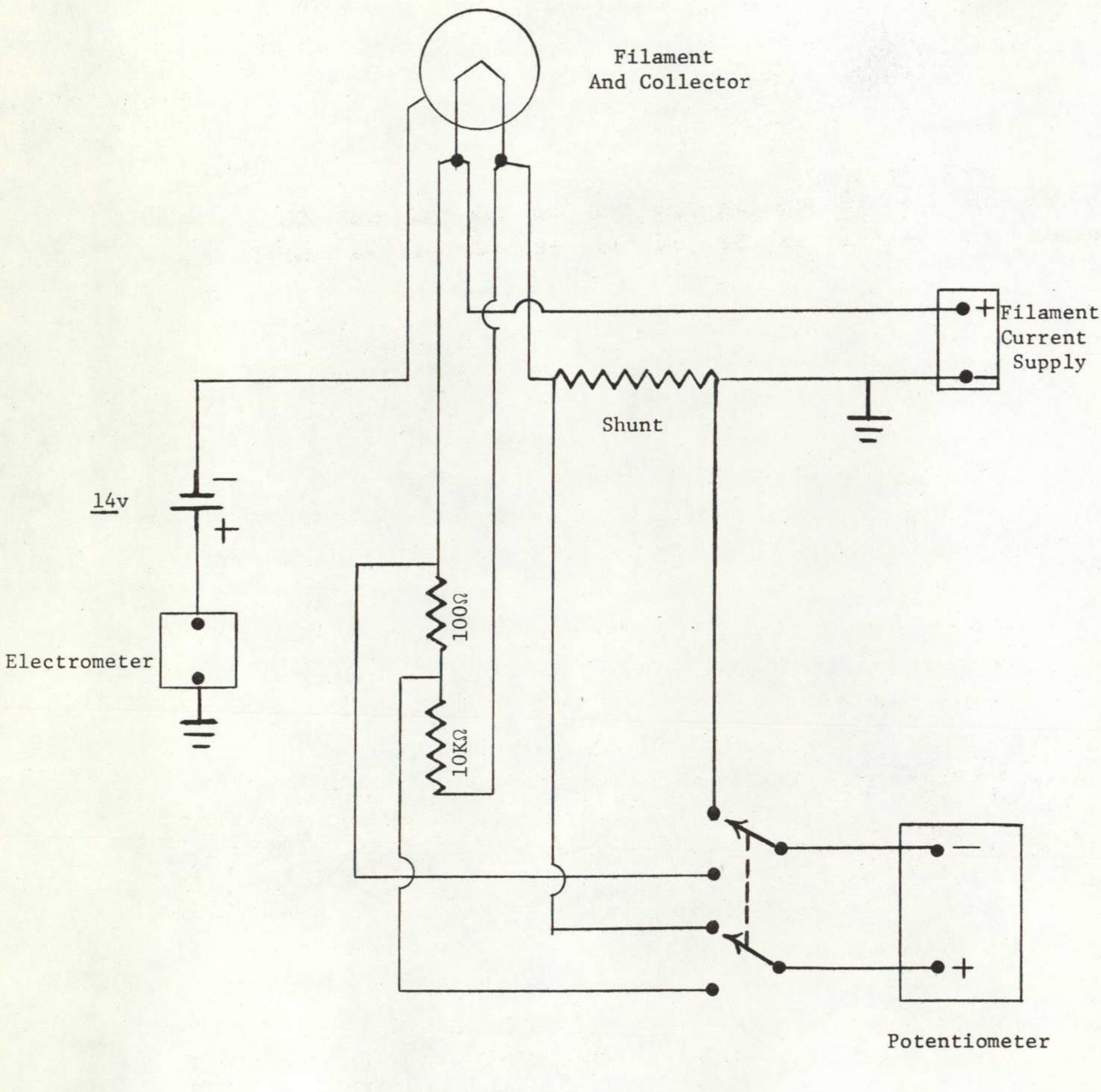


Figure 20. Lithium Fluoride Leak Detector Wiring Schematic Diagram.

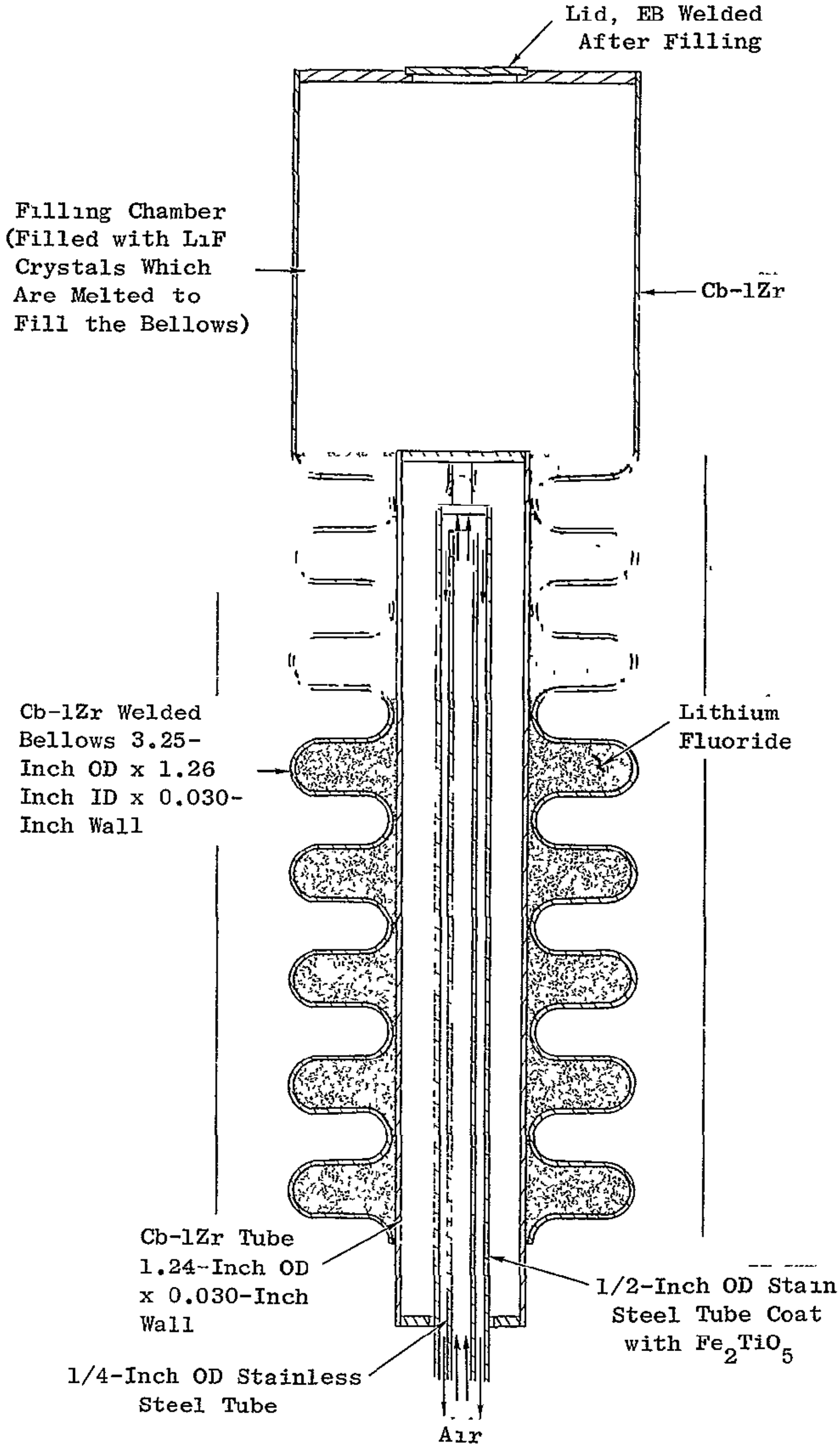


Figure 22. Schematic of Bellows Capsule I Test Facility.

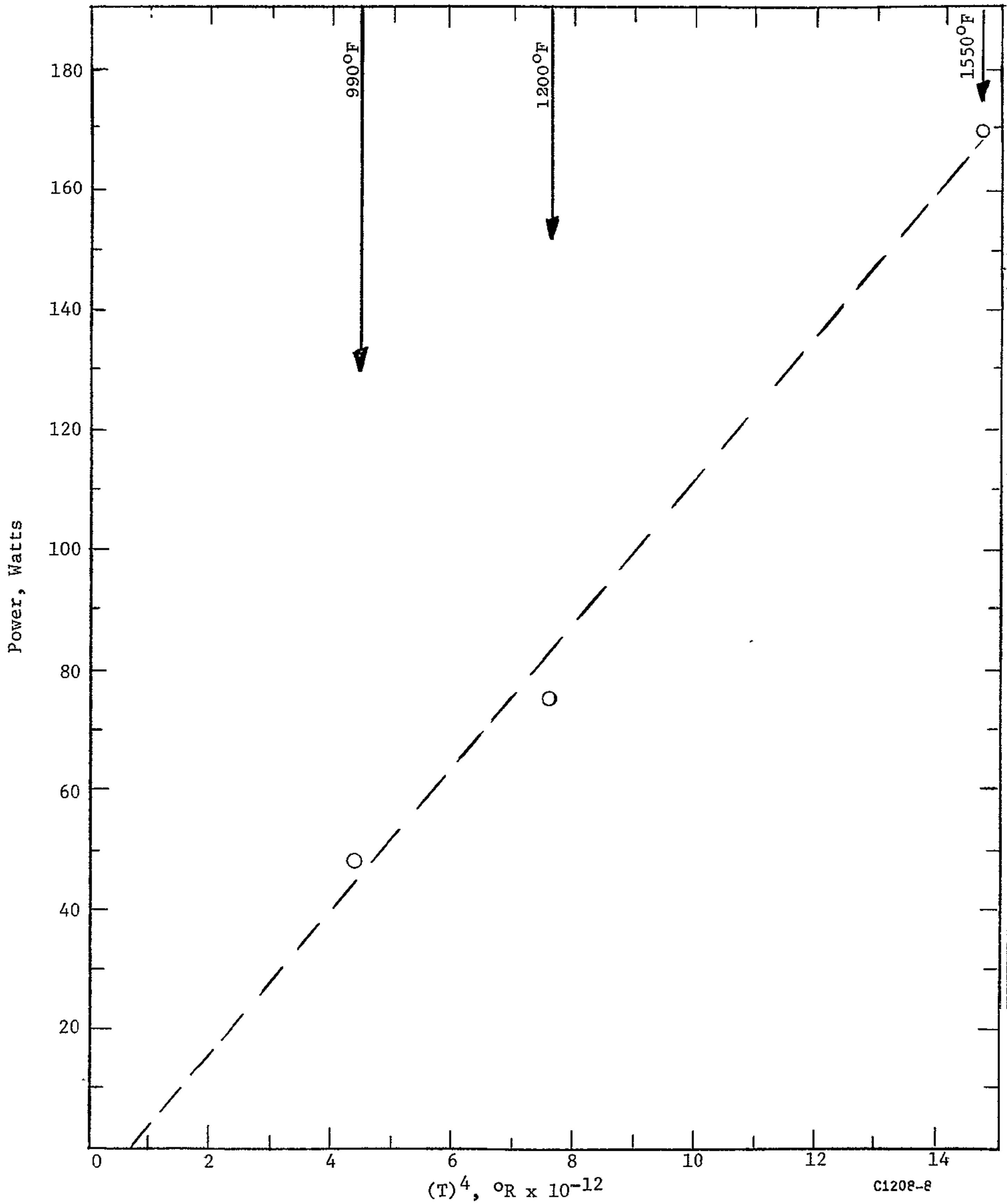


Figure 23. The Steady State Power Temperature Relationship During the Initial Heat-Up of the Lithium Fluoride Bellows Capsule Test. No Air Flowing.

TABLE II.

OPERATING CONDITIONS FOR THE LITHIUM FLUORIDE BELLOWS CAPSULE TEST

Heating	
Time	60 minutes
Maximum Temp.*	1900°F
Current	250 amps
Voltage	3.3 volts
Power	825 watts
Air Flow	11.3 scfm
Chamber Pressure (300th cycle)	1×10^{-8} Torr
Cooling	
Time	36 minutes
Minimum Temp.*	1500°F
Power	(Off)
Air Flow	11.3 scfm

* Measured with the same thermocouple

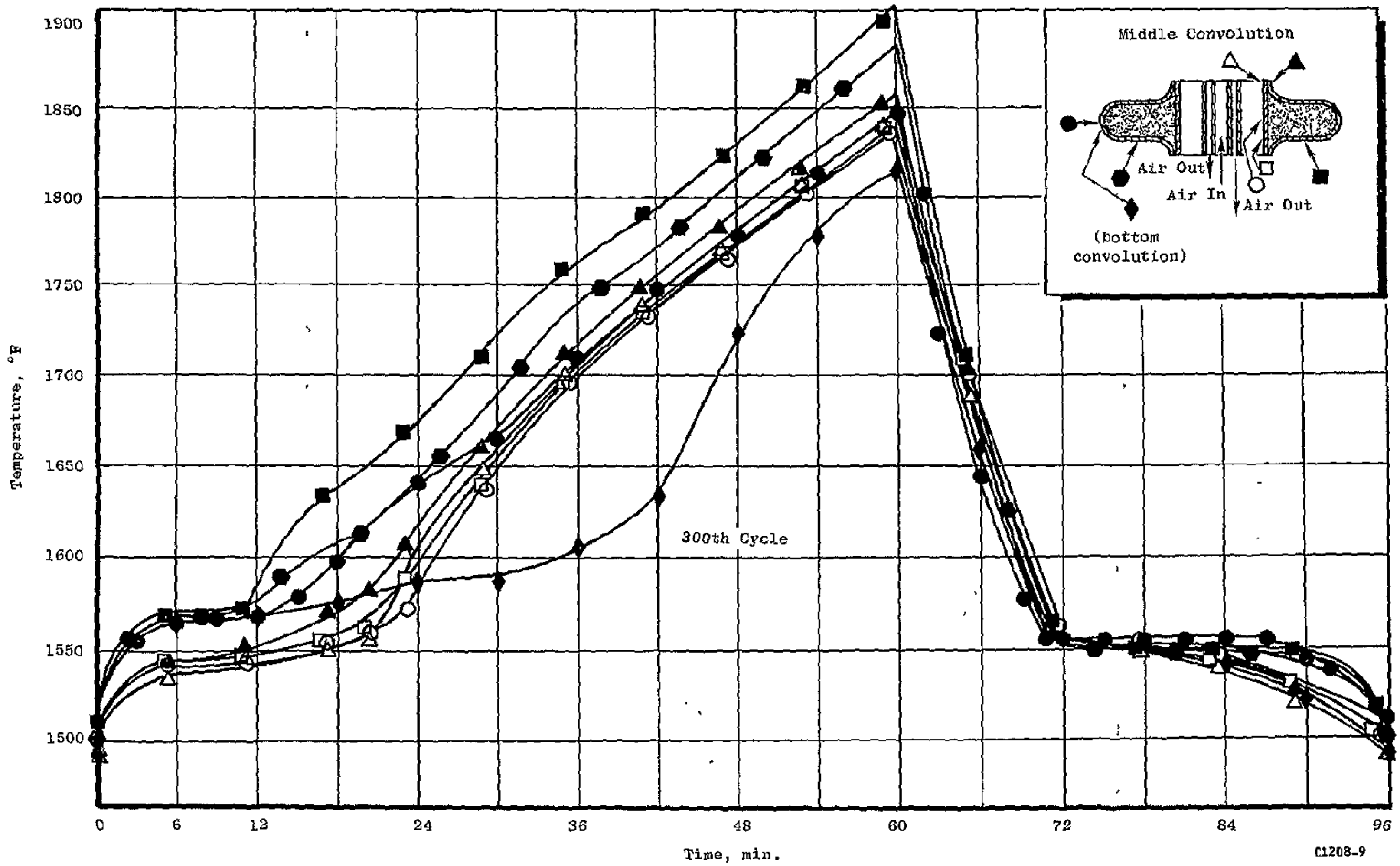


Figure 24. Temperature Profiles on the 300th Cycle of the Lithium Fluoride Bellows Capsule I Test.

conduction losses are negligible at this location. Probably the most important factor in the success of the bellows capsule configuration for containment of lithium fluoride is the lithium fluoride melting-freezing sequence. To compartmentalize the lithium fluoride, thereby providing sufficient void volume to accommodate the large expansion during the heating cycle, the lithium fluoride in the narrow annulus between the inner convolutions and the inner tube must freeze first. The temperature profiles shown in Figure 24 indicate that the last lithium fluoride to melt and first lithium fluoride to freeze in the center convolution is located at the surface of the inner tube. Furthermore, the first lithium fluoride to freeze is located at the 0.010-inch annulus between the inner tube and bellows. This sequence is representative for the lithium fluoride in the bottom convolution as well although the time interval differs as described below.

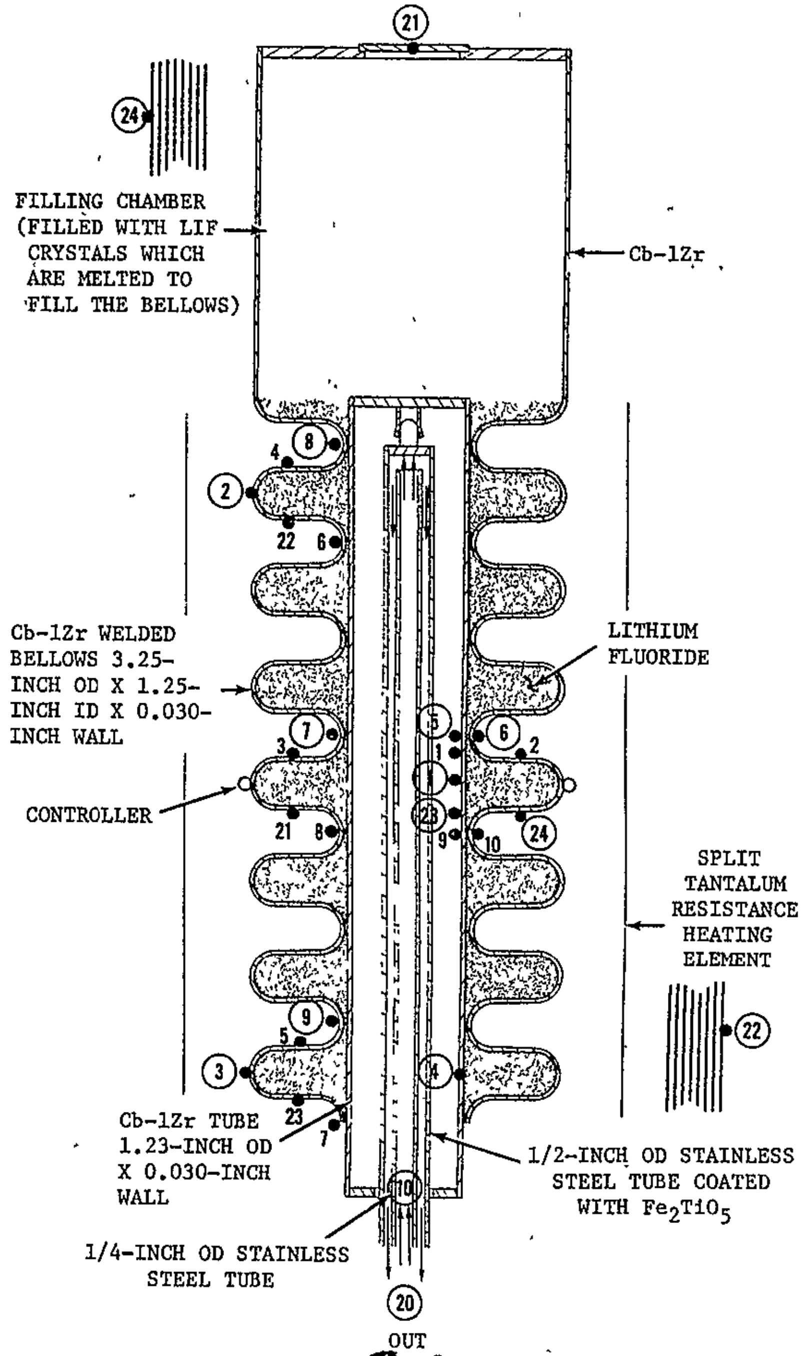
The lithium fluoride in the center convolution cooled from 1900° to 1553°F in 11 minutes, froze in an additional 20 minutes and cooled to 1500°F in 5 minutes. Calculations based upon a heat balance during solidification of the lithium fluoride, presented in Table III, indicate 22 minutes as the required time for freezing which agrees favorably with the thermocouple data illustrated in Figure 24. After completion of 1000 hours corresponding to 625 temperature cycles, the programmed temperature cycling was terminated.

At this time, various thermal measurements were made on the bellows capsule to establish more precisely the heat input and heat extraction during the melting and solidification portions of the thermal cycle.

Steady state temperature measurements were performed at approximate capsule temperatures of 1000°F, 1300°F, 1500°F, and 1600°F with no air flowing through the center cooling tube and with 11.3 scfm of air, the flow rate used during the 1000-hour test, flowing through the center tube. Power, air flow, and temperature measurements were made as described previously. Constant power was applied to the capsule heater for a minimum of 10 hours before steady state temperature measurements were made at each temperature level. Preliminary testing indicated that this time interval was necessary to achieve a steady state. The temperature

TABLE IV. THE STEADY STATE TEMPERATURE PROFILES
LITHIUM FLUORIDE BELLOWS CAPSULE TEST

TEMPERATURE, °F								
Power, Watts	67	115	186	215	109	213	348	444
Thermocouples								
①	1052	1295	1516	1615	1017	1274	1501	1603
②	1034	1274	1492	1592	1010	1270	1515	1608
③	1030	1267	1486	1582	1018	1273	1504	1589
④	1029	1267	1486	1590	997	1241	1459	1569
⑤	1047	1291	1512	1615	1028	1292	1528	1632
⑥	1047	1291	1512	1615	1026	1286	1517	1622
⑦	1049	1291	1511	1615	1031	1292	1511	1616
⑧	1026	1267	1476	1573	995	1258	1476	1583
⑨	1037	1275	1491	1596	1026	1272	1489	1590
⑩	← NO AIR →				65	65	65	65
⑳					90	110	137	154
㉑	811	1024	1216	1311	862	1098	1326	1410
㉒	386	450	581	644	406	536	639	692
㉓	1052	1296	1518	1622	1019	1277	1504	1605
㉔	1049	1293	1516	1621	1035	1304	1544	1645
1	1050	1293	1514	1612	1007	1268	1492	1598
2	1051	1295	1518	1616	1004	1270	1498	1607
3	1050	1293	1514	1616	1037	1300	1534	1636
4	1032	1271	—	1616	1003	1295	1497	1604
5	1032	1269	1486	1590	1012	1275	1499	1590
6	1039	1281	1500	1598	1009	1273	1500	1605
7	1021	1257	1473	1572	993	1238	1456	1550
8	1051	1293	1514	1616	1039	1295	1519	1622
9	1051	1295	1517	1616	1016	1273	1499	1600
10	1045	1287	1509	1612	1018	1278	1506	1610
21	1051	1294	1516	1630	1039	1329	1535	1631
22	1036	1276	1496	1595	1008	1275	1506	1603
23	1027	1263	1480	1582	1007	1259	1486	1574
24	436	547	647	713	458	574	717	772



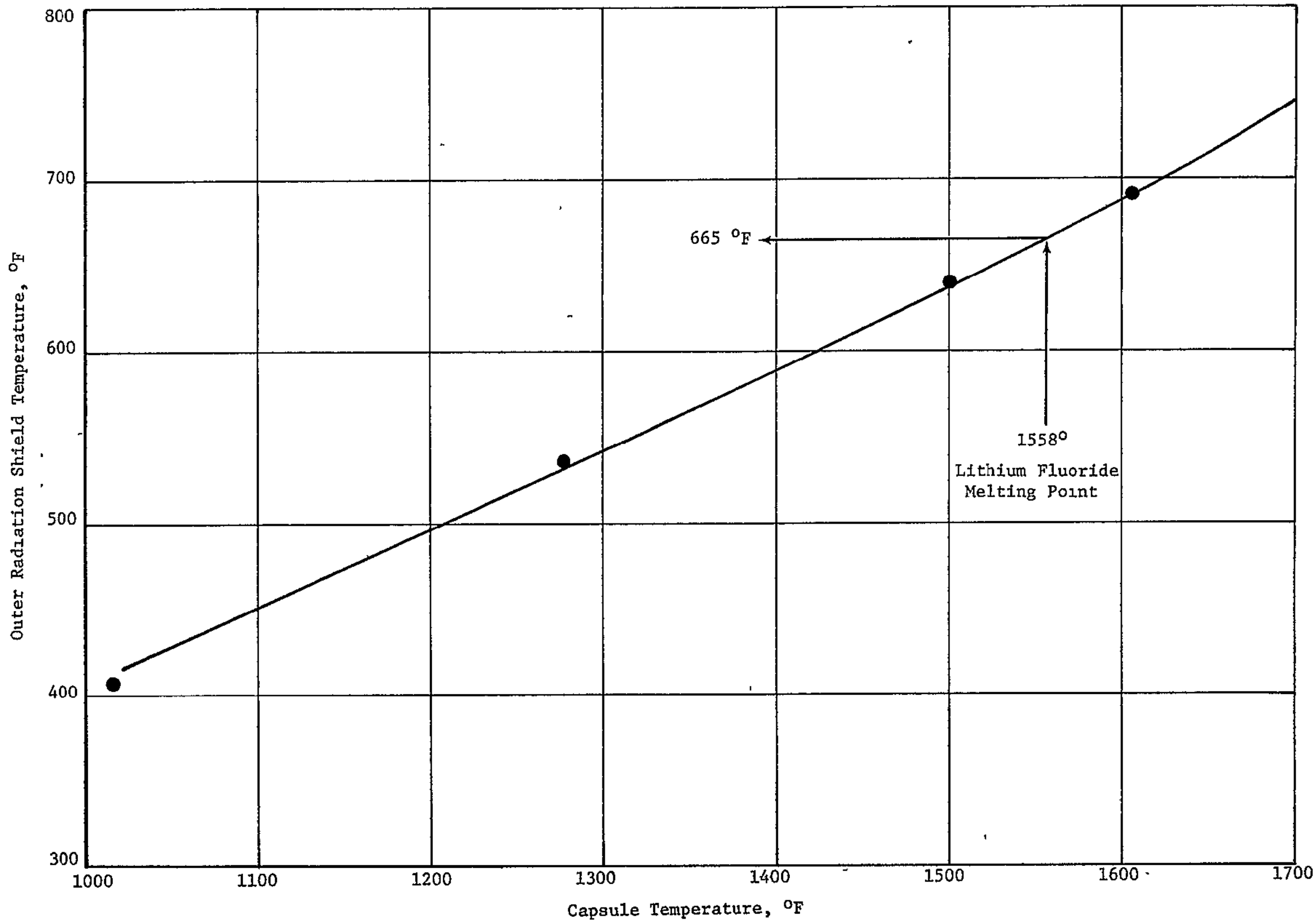


Figure 25. The Steady State Temperature Measurements - Lithium Fluoride Bellows Capsule I Test.

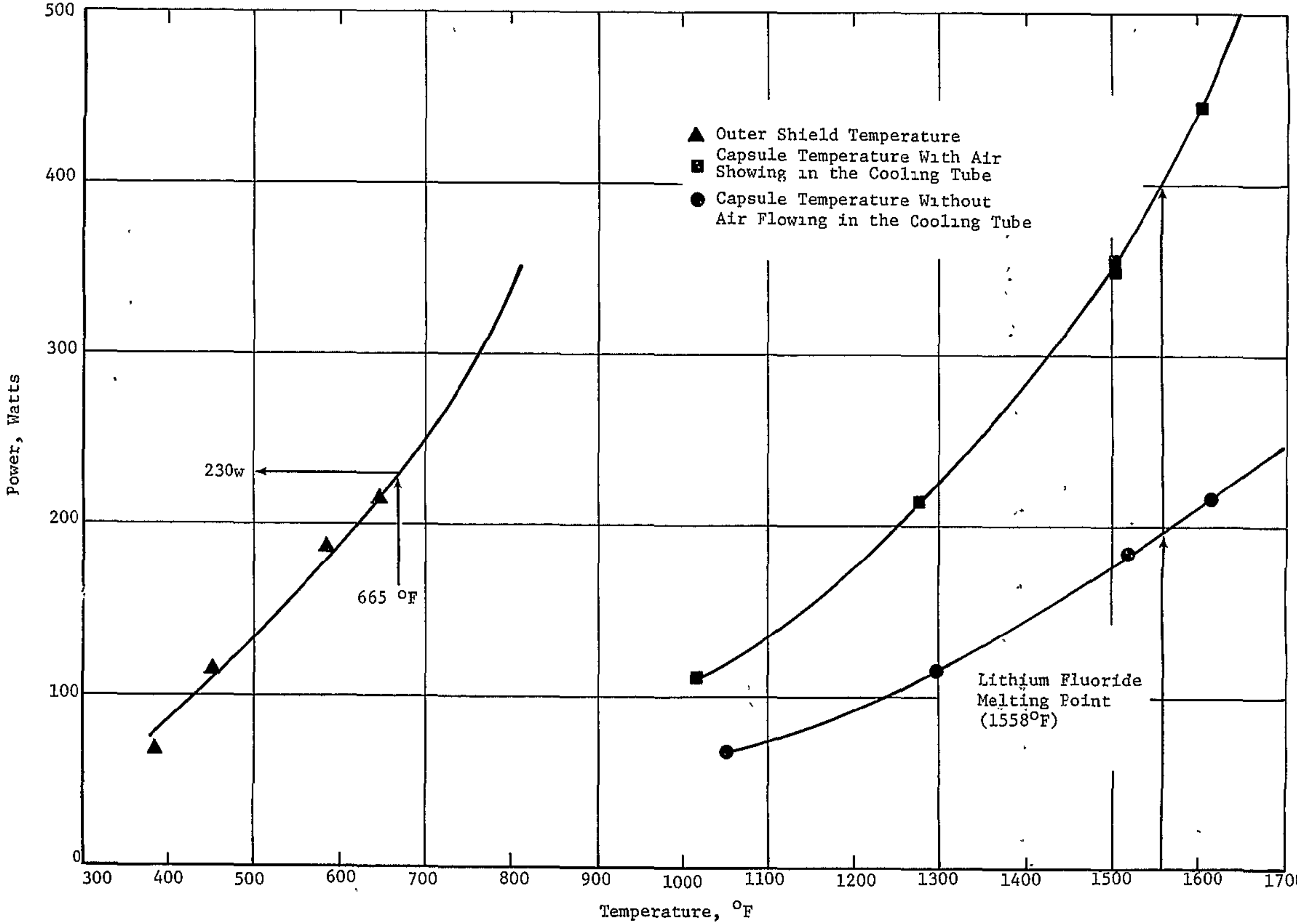


Figure 26. The Steady State Temperature Power Measurements - Lithium Fluoride Bellows Capsule I Test.

TABLE V.

LITHIUM FLUORIDE SOLIDIFICATION TIME
CALCULATED FROM STEADY STATE POWER-TEMPERATURE MEASUREMENTS

Power required to maintain the capsule at 1558°F with 11.3 scfm air flow (Figure 2)	400 watts	(A)
Power loss by radiation based on an outside shield temperature of 665°F	230 watts	(B)
Power rejected to the cooling tube, $\left[A-B\right]$	170 watts or 580 Btu/hr	(C)
Heat of solidification for weight of lithium fluoride in capsule (665 gm x .99 Btu/gm).	685 Btu	(D)

Therefore, time for solidification equals:

$$\left[\frac{D}{C}\right] = \frac{658 \text{ Btu}}{580 \text{ Btu/hour}} = 1.14 \text{ hrs} \times 60 \text{ min/hr} \quad \underline{68.5 \text{ minutes}}$$

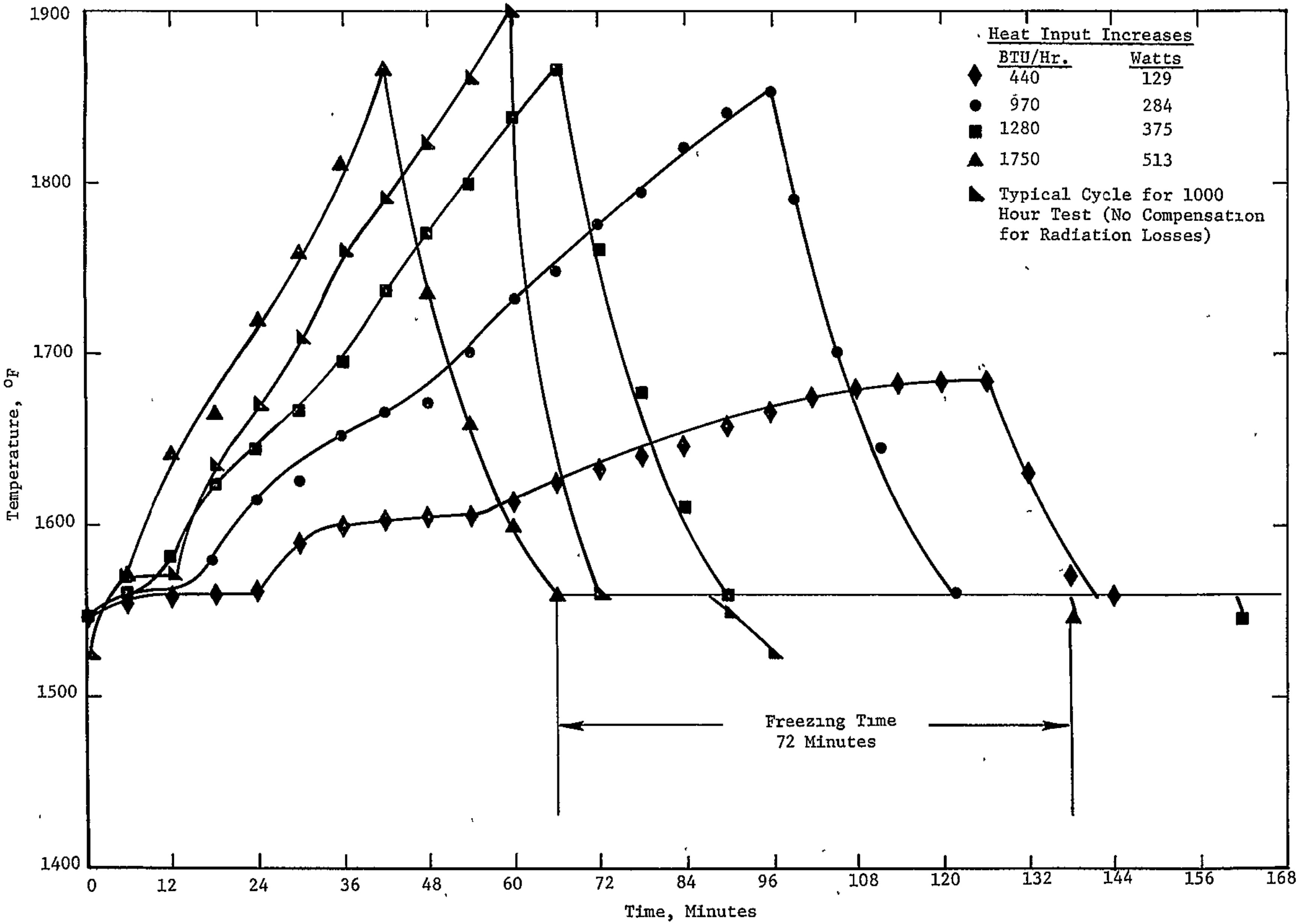


Figure 27. The Heat-up Rates for Various Heat Input Increases Above Steady State Conditions Just Below the Lithium Fluoride Melting Temperature. Radiation Losses are Compensated for on Heating and Cooling.

A typical cycle from the 1000-hour test is shown in Figure 27 for comparison. During this cycle, no compensation was made for radiation losses and as can be seen the cooling rate was more rapid since more than 50% of the heat was lost by radiation through the insulating shields.

After completing these final experiments the bell jar was removed from the test facility. The outer shield assemblies were removed and the capsule was inspected. As can be seen in Figure 28, four of the seven bellows convolutions decreased in thickness (height) during the test. The capsule was removed from the test facility and inspected. The overall capsule height was reduced to 11-1/16 inches as compared to an original height of 12 inches as shown in Figure 29. The cause of the deformation, which is described in detail in the Evaluation Section of this report, is associated with the 1900°F maximum test temperature. As a result the maximum temperature of Bellows Capsule II and III was reduced to 1700°F.

2. Bellows Capsule II

As described previously, Bellows Capsule II was different in design from Bellows Capsule I. Therefore some modifications to the test facility were necessary to accommodate Bellows Capsule II. A schematic at the test setup is shown in Figure 30. The same pumpdown procedure used for Bellows Capsule I was used for Bellows Capsule II. During bakeout the capsule was heated to 600°F to further enhance outgassing of the iron titanate. This was accomplished by supplying additional heat to the capsule with the split tantalum resistance heating element. The chamber heaters were shutdown after 12 hours. At that time a chamber pressure of 2×10^{-8} torr was recorded with the capsule at 600°F and the chamber walls at approximately 100°F. Water cooling of the chamber walls was initiated and the power to the heating element was increased at a rate to maintain the chamber pressure in the 10^{-7} torr range. Prior to thermocycling the capsule, thermal measurements were made on the capsule to establish the radiation losses during the cooling cycle.

a. Steady State Measurements

Steady state temperature measurements were performed at temperatures between 1300°F and 1650°F with no air flowing through the center cooling

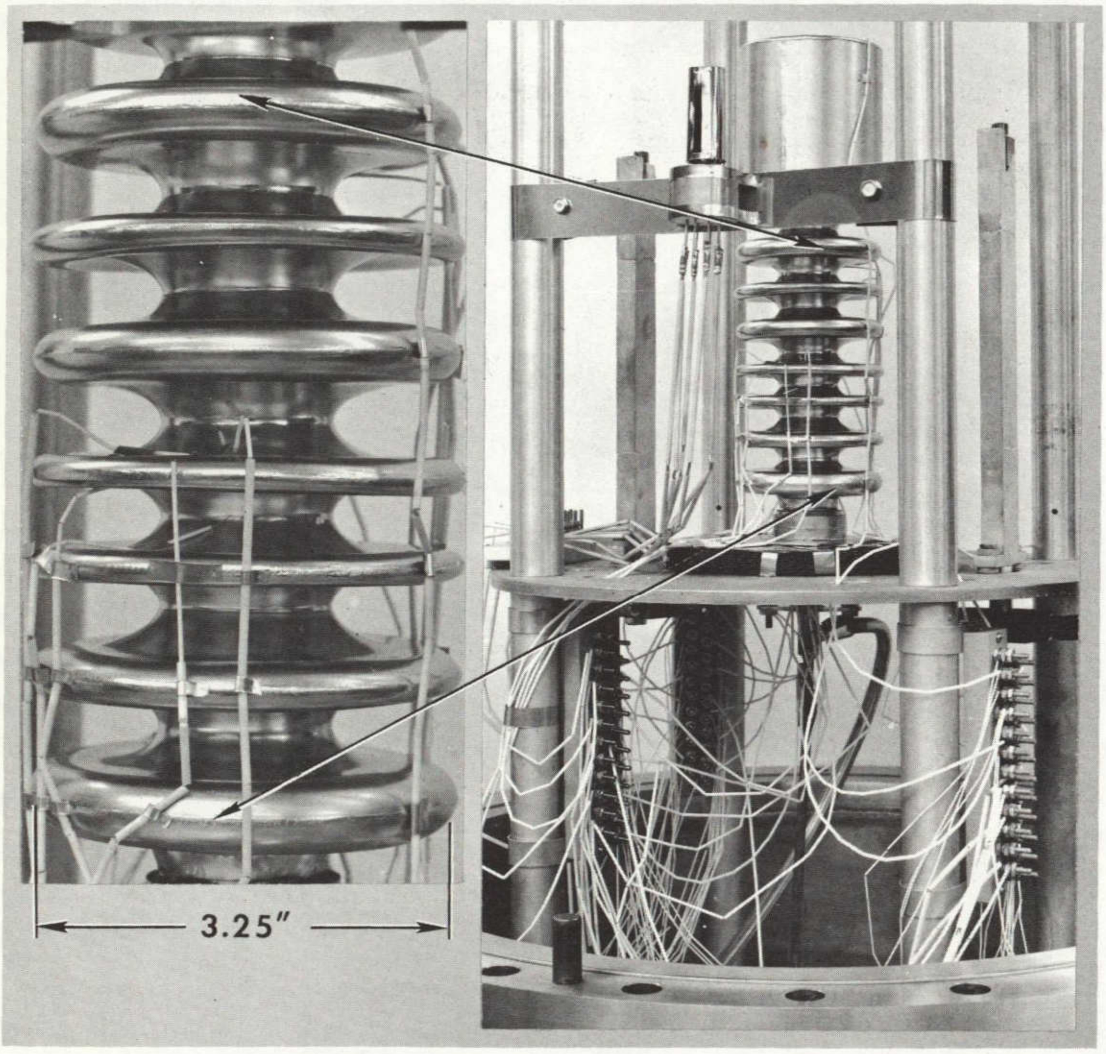
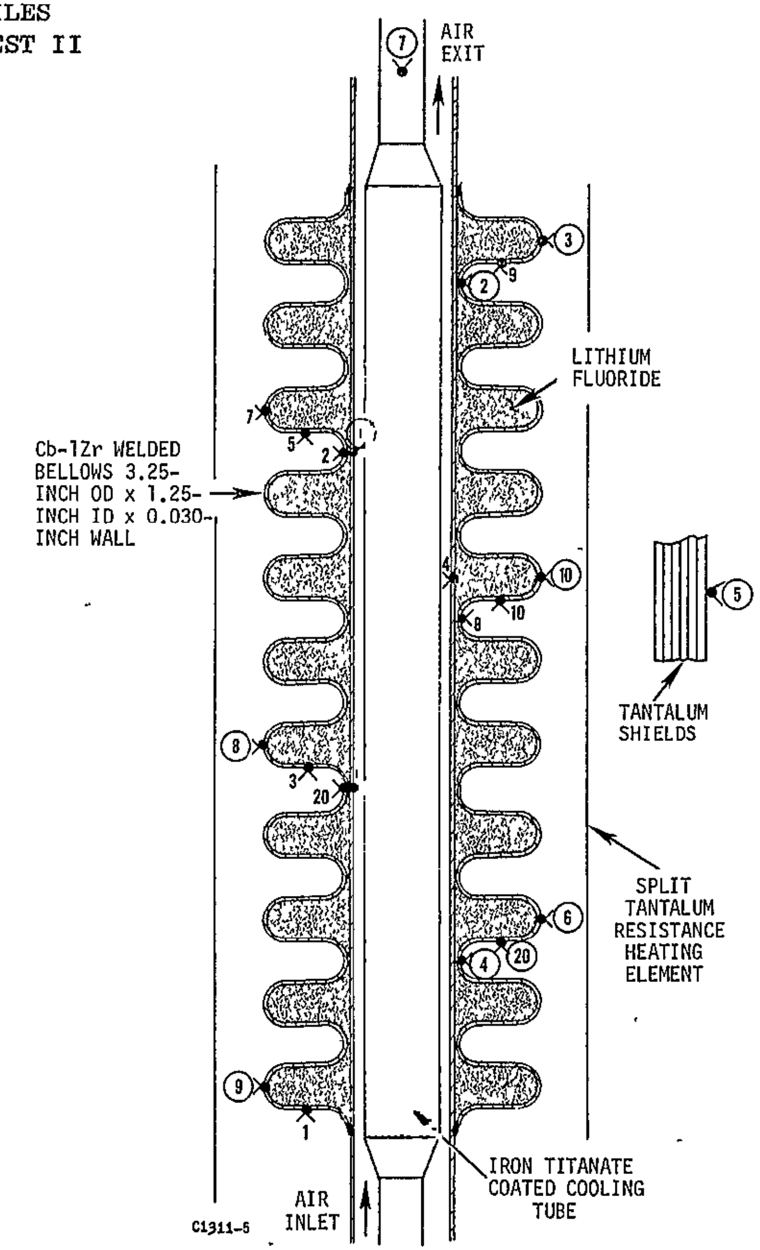


Figure 28. The Lithium Fluoride - Bellows Capsule I After Testing. The Capsule was Cycled 625 Times Between 1520°F and 1900°F in 1000 Hours. (C67041909, C67041910)

TABLE VI. THE STEADY STATE TEMPERATURE PROFILES LITHIUM FLUORIDE BELLOWS CAPSULE TEST II

	TEMPERATURE, °F					
POWER, WATTS	109	163	240	496	684	891
AIR FLOW, SCFM	← NO AIR FLOW →			19	19	19
THERMOCOUPLES						
NUMBERS ENCIRCLED 1	1290	1414	1616	1310	1435	1560
2	1289	1411	1606	1334	1452	1567
3	1282	1404	1601	1379	1498	1587
4	1296	1419	1607	1342	1451	1538
5	330	492	586	535	596	637
6	1339	1468	1663	1430	1556	1614
7	829	908	1042	170	175	208
8	1348	1476	1670	1425	1548	1612
9	1323	1448	1647	1401	1520	1586
10	--	--	--	--	--	--
20	1336	1464	1658	1420	1538	1603
1	1305	1431	1624	1410	1528	1586
2	1324	1450	1645	1353	1476	1582
3	1345	1473	1667	1407	1520	1598
4	1339	1467	1660	1317	1433	1550
5	1324	1449	1643	1393	1517	1597
6	1345	1473	--	--	--	--
7	--	--	--	--	--	--
8	--	--	--	--	--	--
9	1290	1415	1606	1376	1488	1583
10	1341	1467	1660	1393	1514	1592
20	1334	1466	1648	1403	1520	1588
INLET AIR	--	--	--	75	75	75



tube and with 19 scfm of air flowing through the cooling tube. Constant power was applied to the capsule heater for a sufficient time to reduce capsule temperature changes to less than 2°F/hr. This corresponded to a minimum time period of 5 hours. The temperature profiles in the capsule, measured with Leeds and Northrup Potentiometer at each steady state condition, are presented in Table VI.

From the graph of capsule temperature* with air flowing in the cooling tube vs. the outer radiation shield temperature, shown in Figure 31, a shield temperature of 600°F can be found to correspond to a capsule temperature at the melting point of lithium fluoride. From the power-temperature curve for the outer radiation shield, shown in Figure 32, a power value of 250 watts can be found to correspond to an outer shield temperature of 600°F. Neglecting small conduction losses, all the heat loss must occur by radiation when no air is flowing in the cooling tube. The power input, when the capsule is at steady state with no air flowing in the cooling tube, can therefore be equilibrated to radiation loss. Using Figures 31 and 32 it may be concluded that the radiation loss is equivalent to 250 watts when the capsule is at 1558°F. Application of 250 watts to the split tantalum heater during the cooling portion of the cycle compensates for this radiation loss. Under this condition capsule cooling occurs by heat rejection to the center cooling tube, a better simulation of the heat rejection mode in the Solar Brayton Cycle Heat Receiver.

b. Test Initiation

During the initial thermal cycling of Bellows Capsule II adjustments were made to the cam and current settings to establish the proper cycle. The operating conditions on the 16th cycle are shown in Table VII. The heat balance for the heating and cooling cycles is presented in Table VIII. The temperature profile of a typical convolution during the 16th cycle is presented in Figure 33. The experimental freezing time of approximately 26 minutes agrees favorably with the calculated time of

*Thermocouple 8 (Table VI) was selected to represent the capsule temperature.

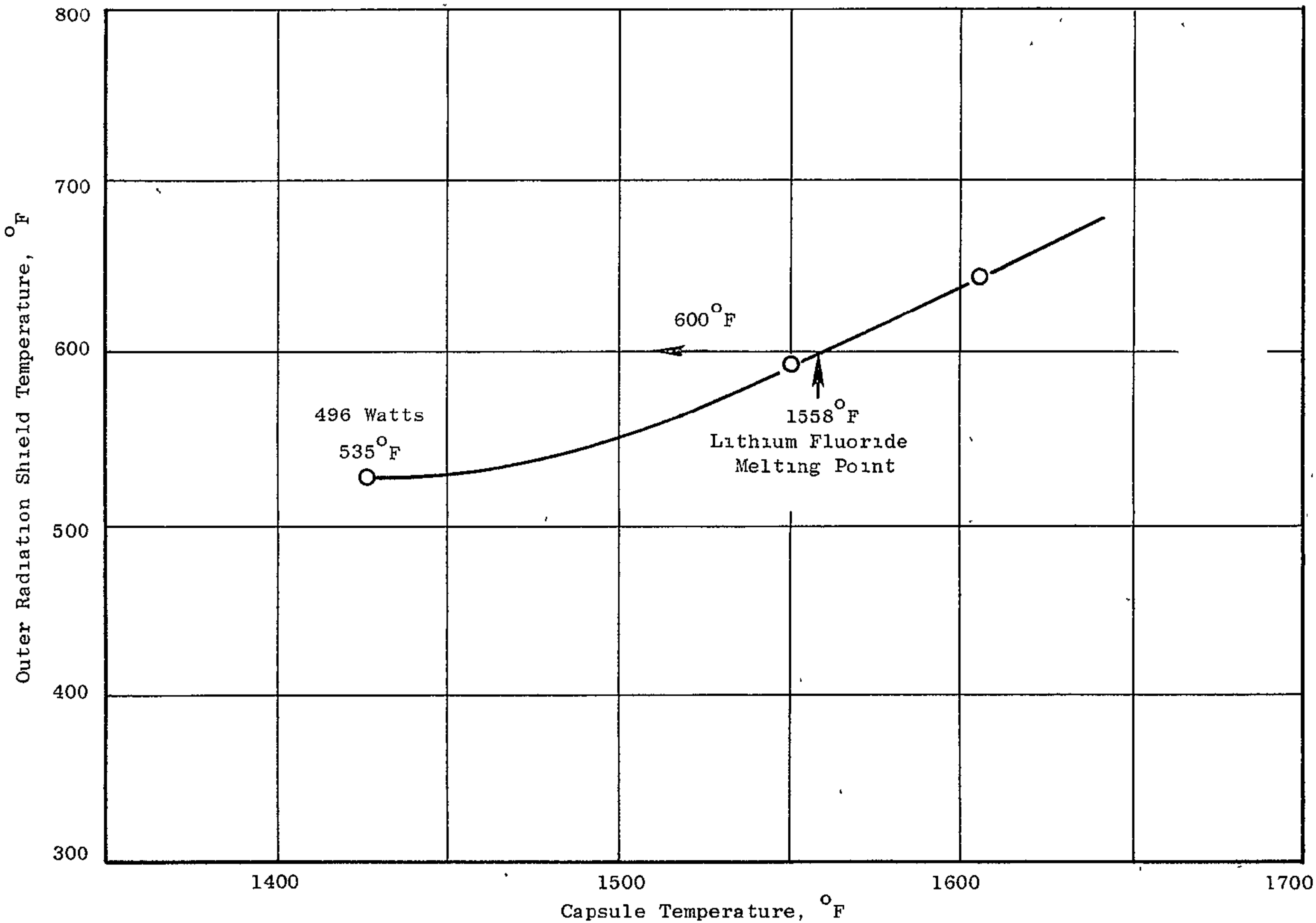


Figure 31. The Steady State Temperature Measurements with 19SCFM Air Flowing in the Cooling Tube - Lithium Fluoride Bellows Capsule Test II.

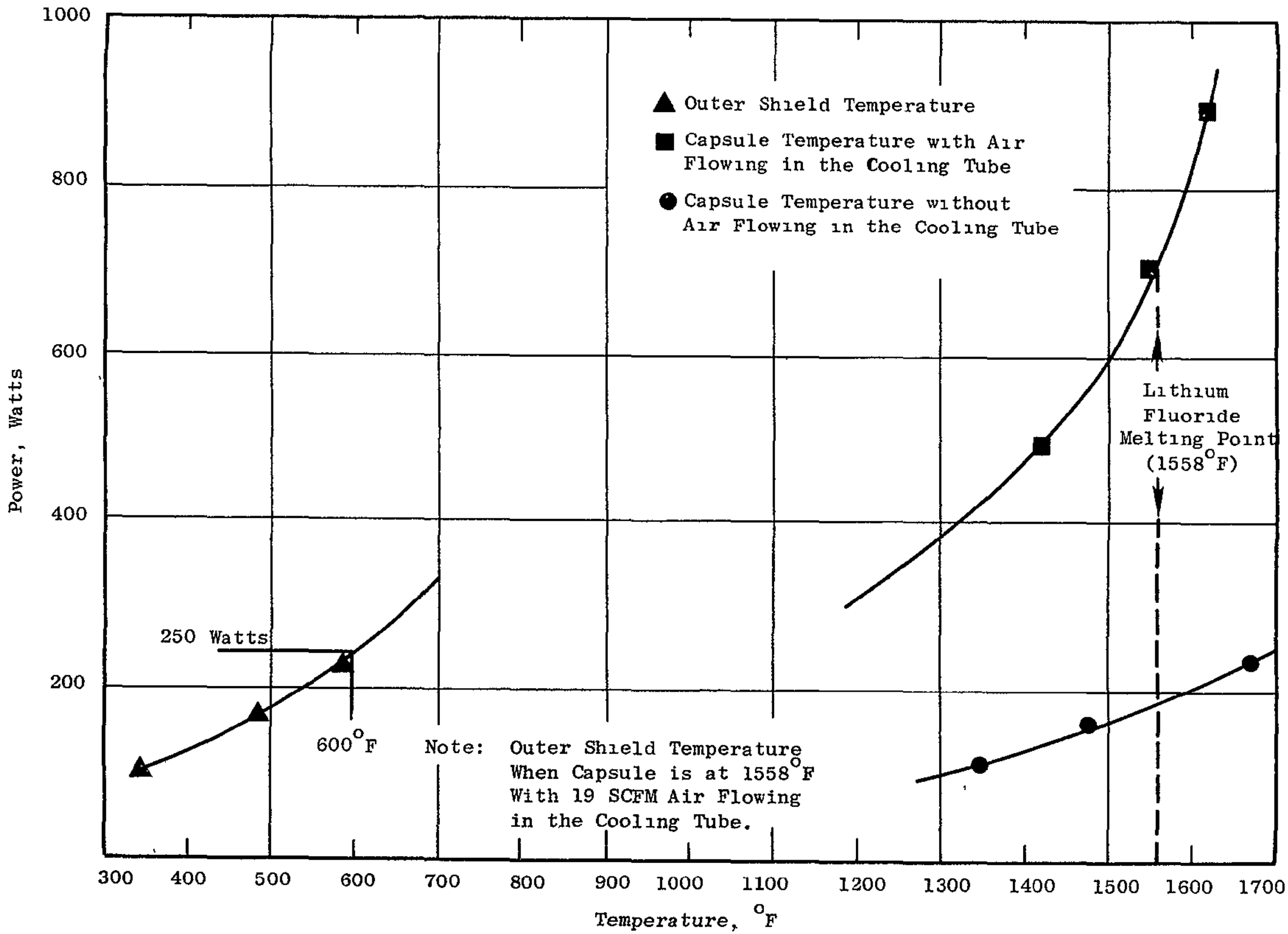


Figure 32. The Steady State Temperature Power Measurements - Lithium Fluoride Bellows Capsule Test II.

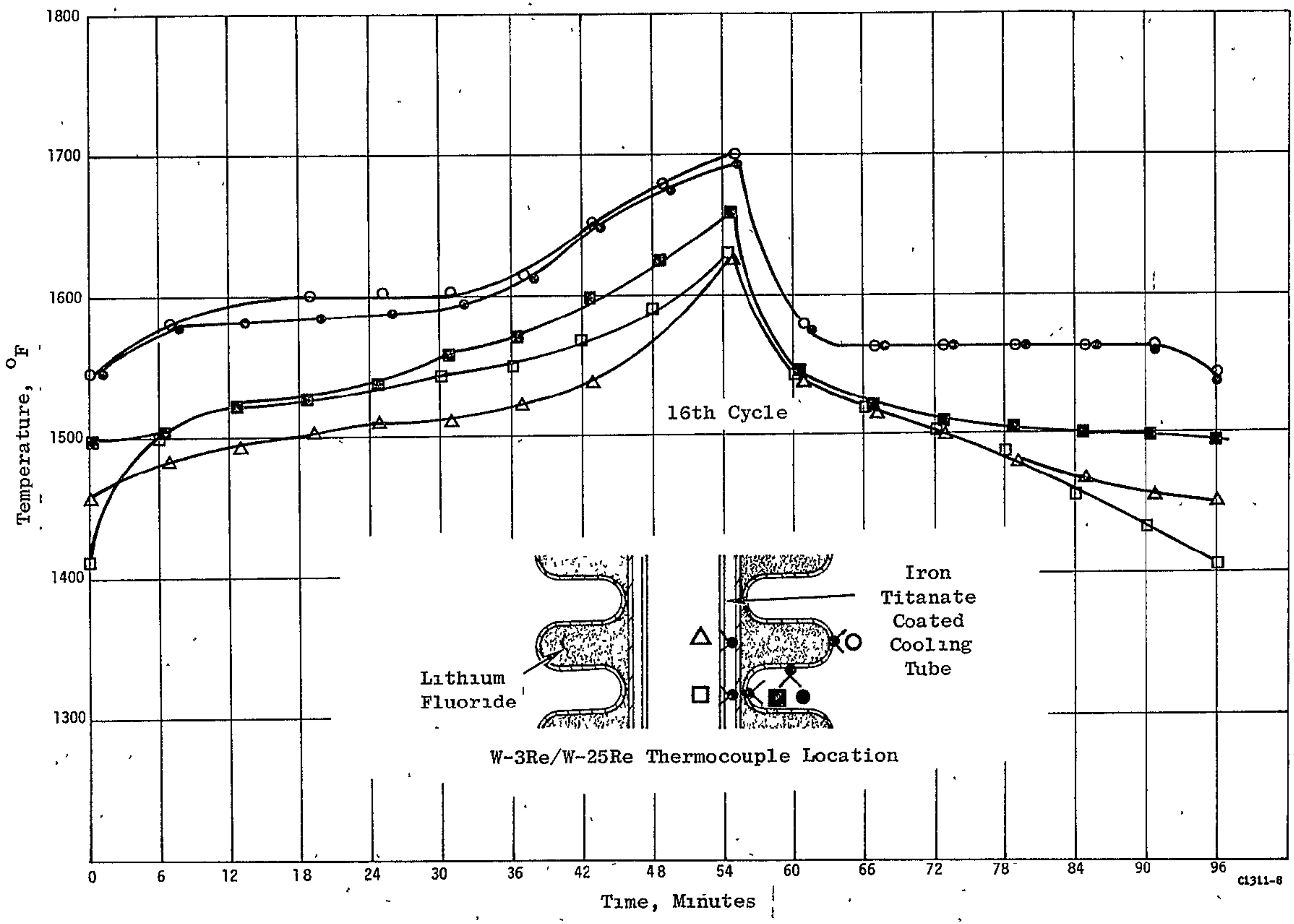


Figure 33. Temperature Profiles on the 16th Cycle of the Lithium Fluoride Bellows Capsule Test II.

TABLE VII.

OPERATING CONDITIONS FOR THE LITHIUM
FLUORIDE BELLOWS CAPSULE TEST II - 16th CYCLE

HEATING	Time	55 minutes
	Maximum Temperature*	1700°F
	Current	330 amps
	Voltage	4.1 volts
	Total Power	1355 watts
	Air Flow	19 scfm
	Chamber Pressure	6×10^{-8} torr
COOLING	Time	41 minutes
	Minimum Temperature*	1545°F
	Power	250 watts
	Air Flow	19 scfm
	Chamber Pressure	4×10^{-8} torr

* Measured with the same thermocouple.

TABLE VIII.

HEAT BALANCE - LITHIUM FLUORIDE BELLOWS CAPSULE TEST II

Since:	Weight of Lithium Fluoride in Capsule	1140 grams
	Molecular Weight of Lithium Fluoride.	25.94 g/moles
	Moles of Lithium Fluoride in Capsule	44 moles
	Heat of Solidification per mole	25.7 Btu
	Total Heat of Solidification.	1130 Btu

FUSION OF LITHIUM FLUORIDE

A.	Total Power.	1355 watts
B.	Power Needed to Maintain 1550°F[a].	680 watts
C.	Total Power to Melt Lithium Fluoride (A-B)	675 watts
D.	Radiation Loss at 1550°F[b].	240 watts
E.	Power to Melt Lithium Fluoride (C-D)	435 watts
	. . . .	(1485 Btu/hr)

Time Required to Melt the Lithium Fluoride

$$\frac{1130 \text{ Btu}^{(c)}}{1485 \text{ Btu/hr}} \times 60 \text{ min/hr} = \underline{45 \text{ minutes}}$$

SOLIDIFICATION OF LITHIUM FLUORIDE

F.	Power to Compensate for Radiation Loss[b].	250 watts
G.	Heat Dissipated by Cooling Tube at 1558°F with 19 scfm Air Flow and 137°F ΔT between Inlet and Exit Air	2830 Btu/hr

Time Required to Solidify the Lithium Fluoride

$$\frac{1130 \text{ Btu}^{(c)}}{2830 \text{ Btu/hr}} \times 60 \text{ min/hr} = \underline{24 \text{ minutes}}$$

(a) Obtained from Figure 32

(b) Obtained from Figures 31 and 32

(c) Heat of solidification

24 minutes based on the heat balance (Table VIII).

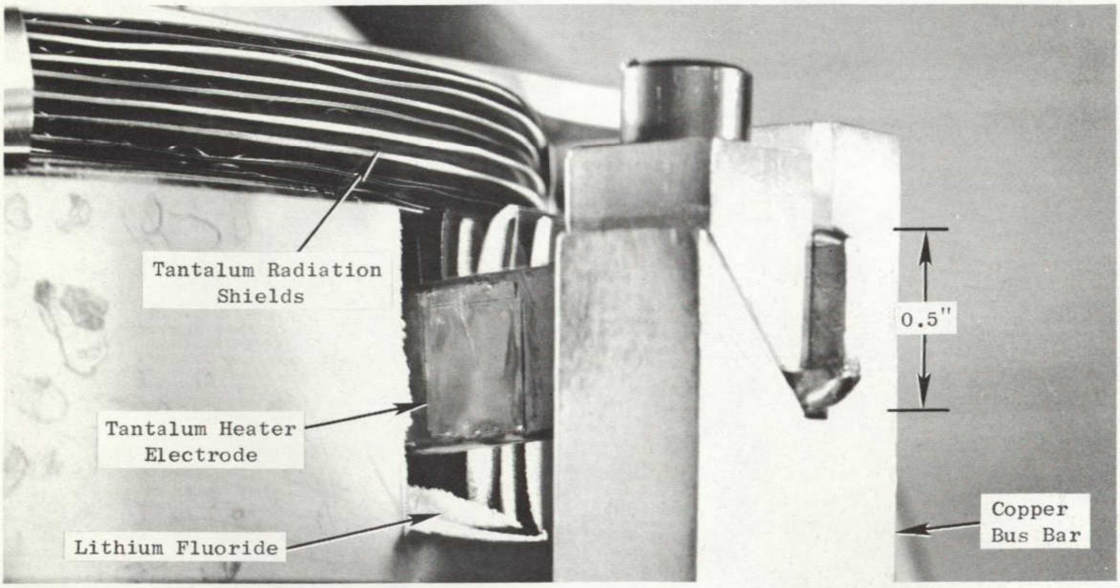
The plan was to test Bellows Capsule II for a total of 5000 hours corresponding to 3125 thermal cycles with intermediate inspections after 1000 hours, and 3000 hours of testing. The capsule was not removed from the test facility during these inspections. Dimensional and radiographic examinations were performed with the capsule in situ. On completion of the 625th cycle (1000 hrs) and 1875th cycle (3000 hrs), the power to the split tantalum heater was kept at 250 watts, the power required to compensate for radiation losses, until the capsule temperature reached 1400°F. At that temperature, the power was shut off and the capsule allowed to cool to room temperature with air flowing in the cooling tube. This cool down procedure was employed to simulate the cool down in the Brayton Cycle Receiver and thereby insure a similar freezing pattern.

Visual inspection of the capsule after 1000 hours test time revealed no significant change in the capsule. Some discoloration of the iron titanate coating was noted on the bottom two convolutions as shown in Figure 34; however the coating's adherence did not seem affected and no significant changes in capsule dimensions were noted.

Visual examination of the test assembly, following removal of the vacuum chamber bell jar, after 3000 hours test tube revealed a deposit of lithium fluoride on the tantalum radiation shields and the copper bus bars as illustrated in Figure 35. Additional lithium fluoride deposits were found on the cooling tube and thermocouple insulators following removal of the tantalum shielding as shown in Figures 36 and 37. Visual inspection of the capsule itself did not indicate any significant changes in shape or appearance as can be seen in Figure 38. The capsule was removed from the test facility and examination continued.

Removal of the tantalum shielding on the lower fill tube revealed a glassy transparent deposit of solidified lithium fluoride. This deposit, shown in Figure 39 (a) differed in morphology from the vapor deposits previously shown and is indicative of solidified liquid lithium fluoride. The suspect tube was cleaned of lithium fluoride, and upon closer inspection, was found to be cracked in the weld area as shown in Figure 39 (b). The fill tube was cut from the capsule and cleaned of

Figure 34. Bottom Convolutions of Lithium Fluroide Bellows Capsule II Following 1000 Hours of Testing Showing Discoloration of the Coating. The Capsule was Cycled Between 1550°F and 1700°F. (C67121173)



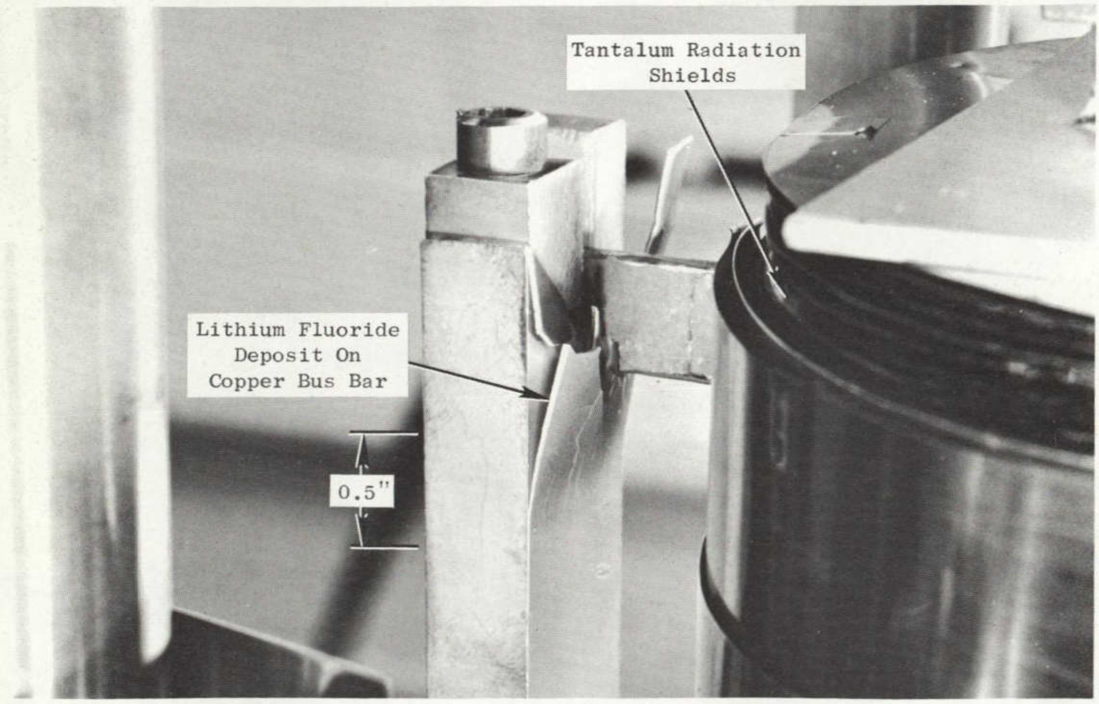


Figure 35. Lithium Fluoride Bellows Capsule II Test Facility After 3000 Hours of Testing Showing Lithium Fluoride Deposits on Copper Bus Bars and Tantalum Radiation Shields. (69031980 & C68031830)

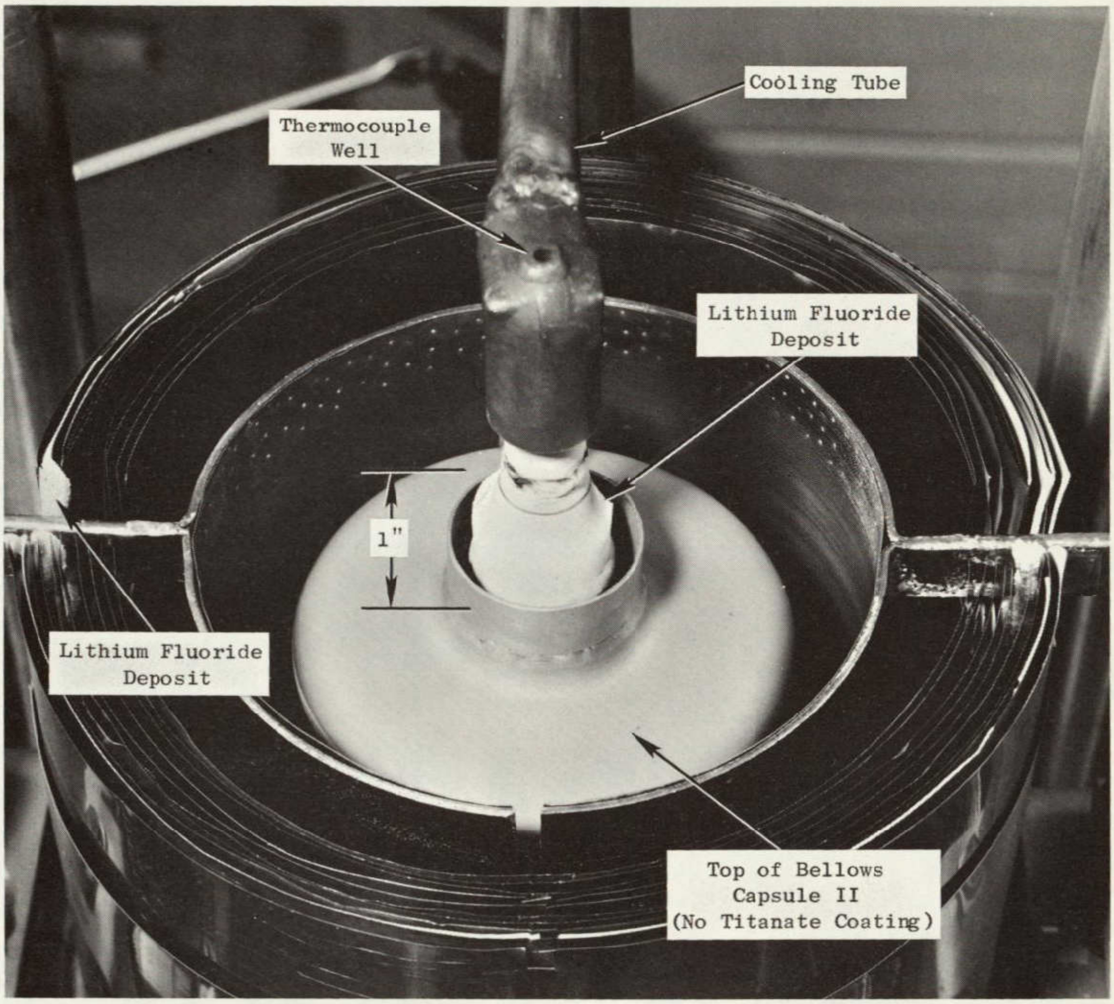


Figure 36. Lithium Fluoride Bellows Capsule II After 3000 Hours of Testing Showing Lithium Fluoride Deposits on Cooling Tube and Tantalum Shielding. (C68031821)

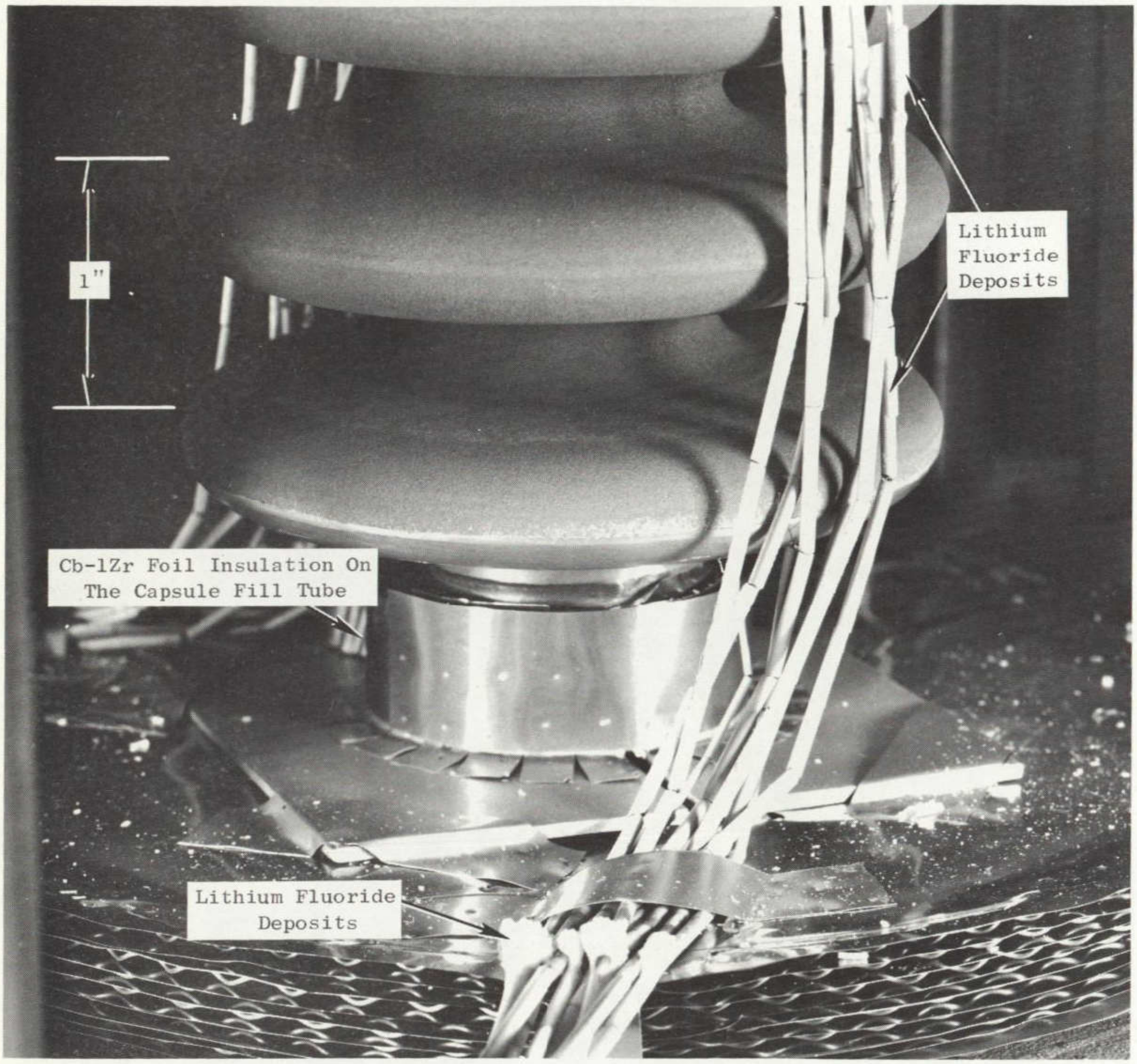


Figure 37. Lithium Fluoride Bellows Capsule II After 3000 Hours of Testing Showing Lithium Fluoride Deposits on Thermocouple Insulators. (C68031820)

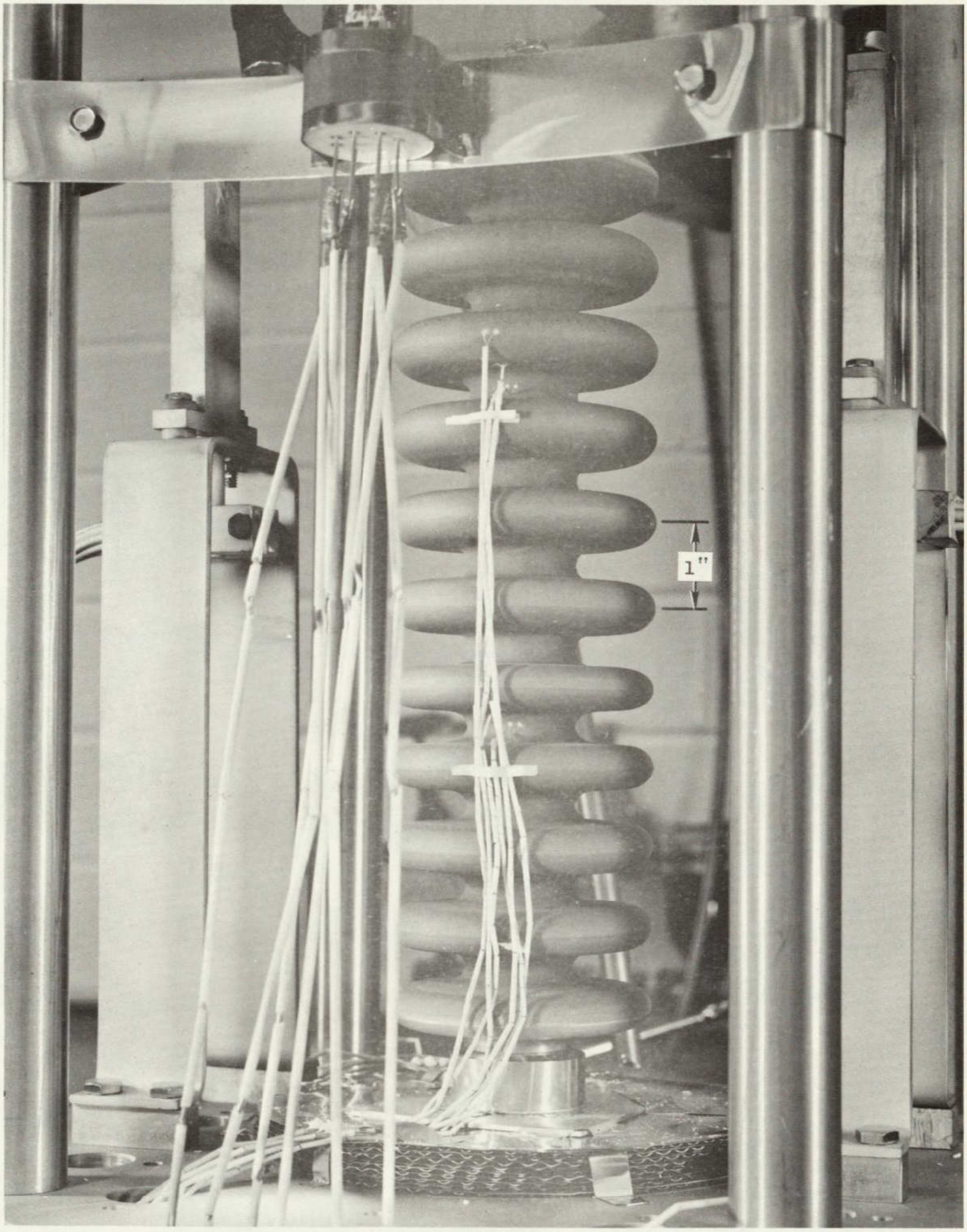
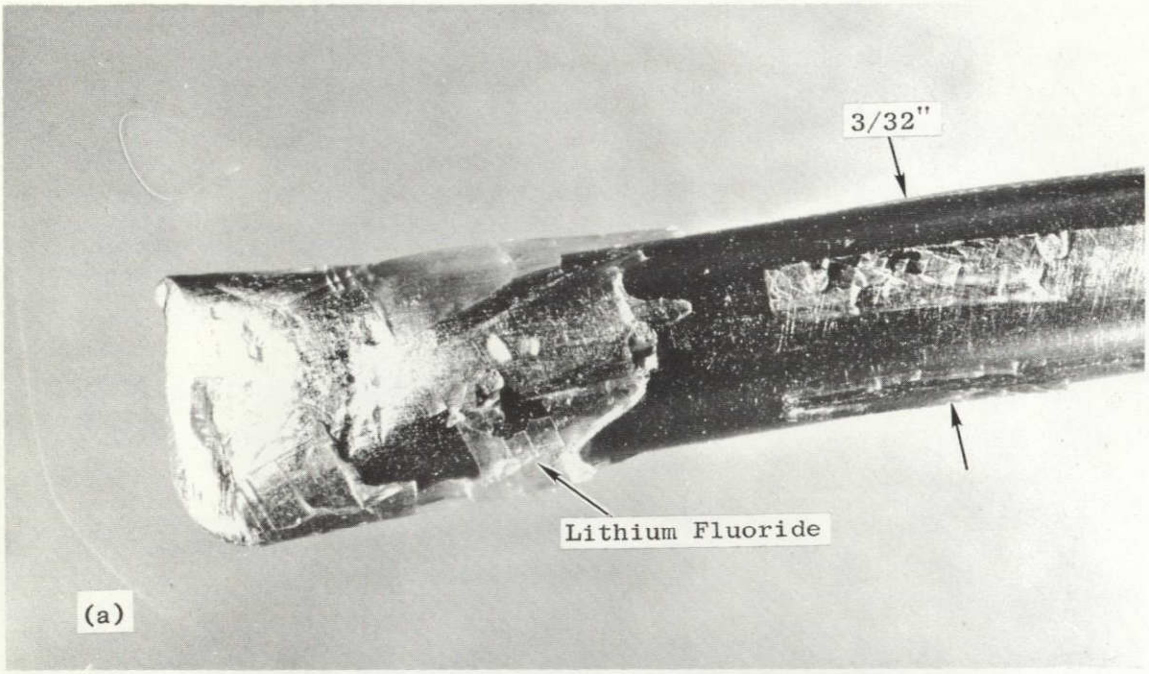


Figure 38. Lithium Fluoride Bellows Capsule II After 3000 Hours of Testing. (C68031822)



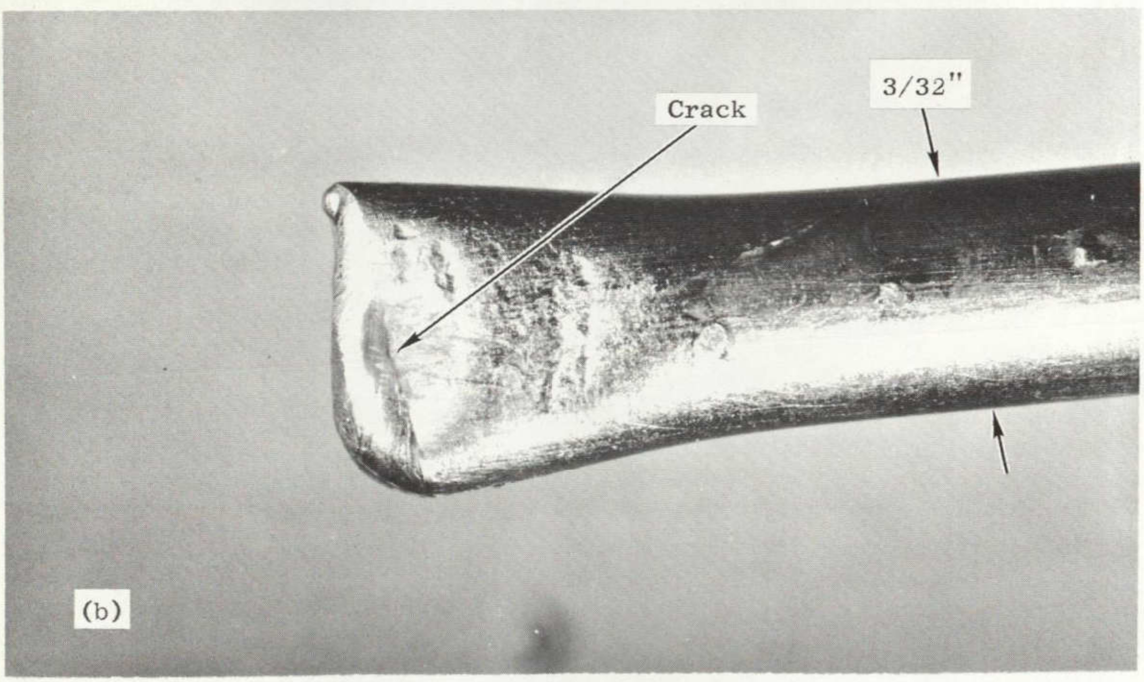


Figure 39. Lower Fill Tube of Bellows Capsule II After Completion of 3000 Hour Test Before (a) and Following Removal (b) of the Lithium Fluoride. Leakage of Lithium Fluoride Occurred at the Indicated Crack.
(a) C68031983 & (b) C69031926

lithium fluoride by boiling water. Subsequent helium mass spectrometric leak checking revealed the crack to be the source of the leak. Photomicrographs of the cracked area are shown in Figure 40. The crack is in the heat affected zone adjacent to the nugget of the closure weld. The appearance of the matrix adjacent to the crack and at the ID suggests the Cb-1Zr reacted with lithium fluoride present in this region during welding.

After the capsule was removed from the test facility, it was weighed revealing 4.5 grams or only 0.3% by weight of the lithium fluoride originally in the capsule had leaked out. Any loss in lithium fluoride volume is not readily discernable by comparison of radiographs of the capsule before and after test as shown in Figure 41.

The lithium fluoride leak detector was inoperative at the time the leak occurred as a result of an open filament which occurred after 1200 hours of testing. No pressure rises of an unknown nature were noted during testing which might have indicated a leak. This is understandable since no lithium fluoride deposits were found on the vacuum chamber walls. During the last 2000 hours at testing the pressure remained in the 10^{-8} torr range, varying between 6×10^{-8} and 8×10^{-8} torr at the low and high temperature points of the cycle respectively.

Temperature data were plotted from cycles at approximately 75-cycle intervals, throughout the last 2000 hours of testing showed no significant changes which would indicate capsule deformation or loss of lithium fluoride.

Dimensional data obtained after 3000 hours of operation along with data obtained before test and after 1000 hours of operation are presented in Table IX. A very small increase in the diameter of a number of convolutions can be noted after 3000 hours of testing. An evaluation of the increases noted on the diameter of convolutions 2-10 (end convolutions not considered of end effects) reveals an average diametral growth of 4 mils. The maximum growth noted was 8 mils and was noted on convolutions number 4, 6 and 7.

Examination of the iron titanate coating on Bellows Capsule II after 3000 hours of testing indicated significant changes in appearance as can be seen in Figure 42.

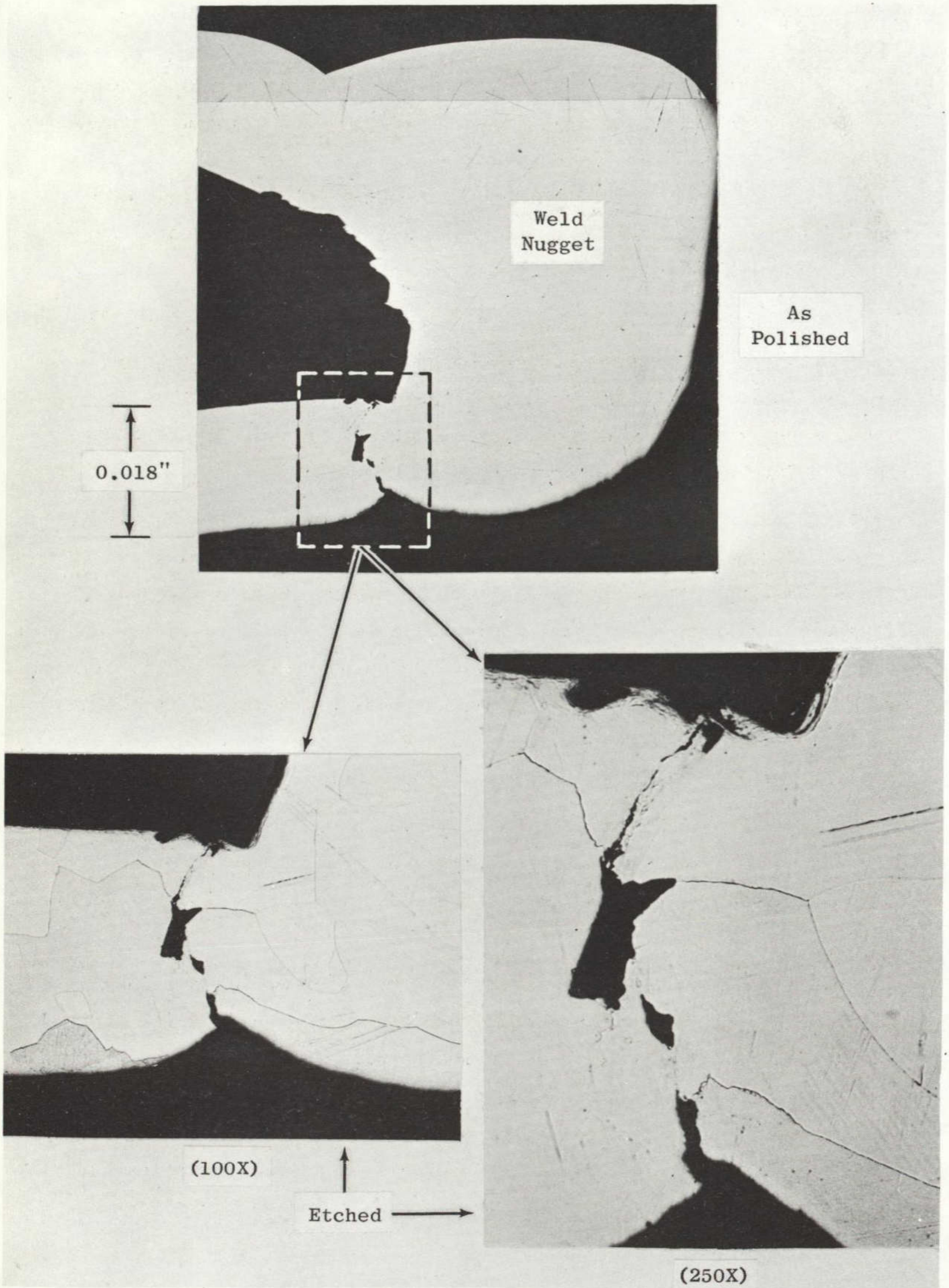


Figure 40. Photomicrographs of the Crack in the Lower Fill Tube Which Caused Lithium Fluoride to Leak from Bellows Capsule II. (F650115) (F650112)
Etchant: 60 Glycerine, 20HNO_3, 20HF (F650114)

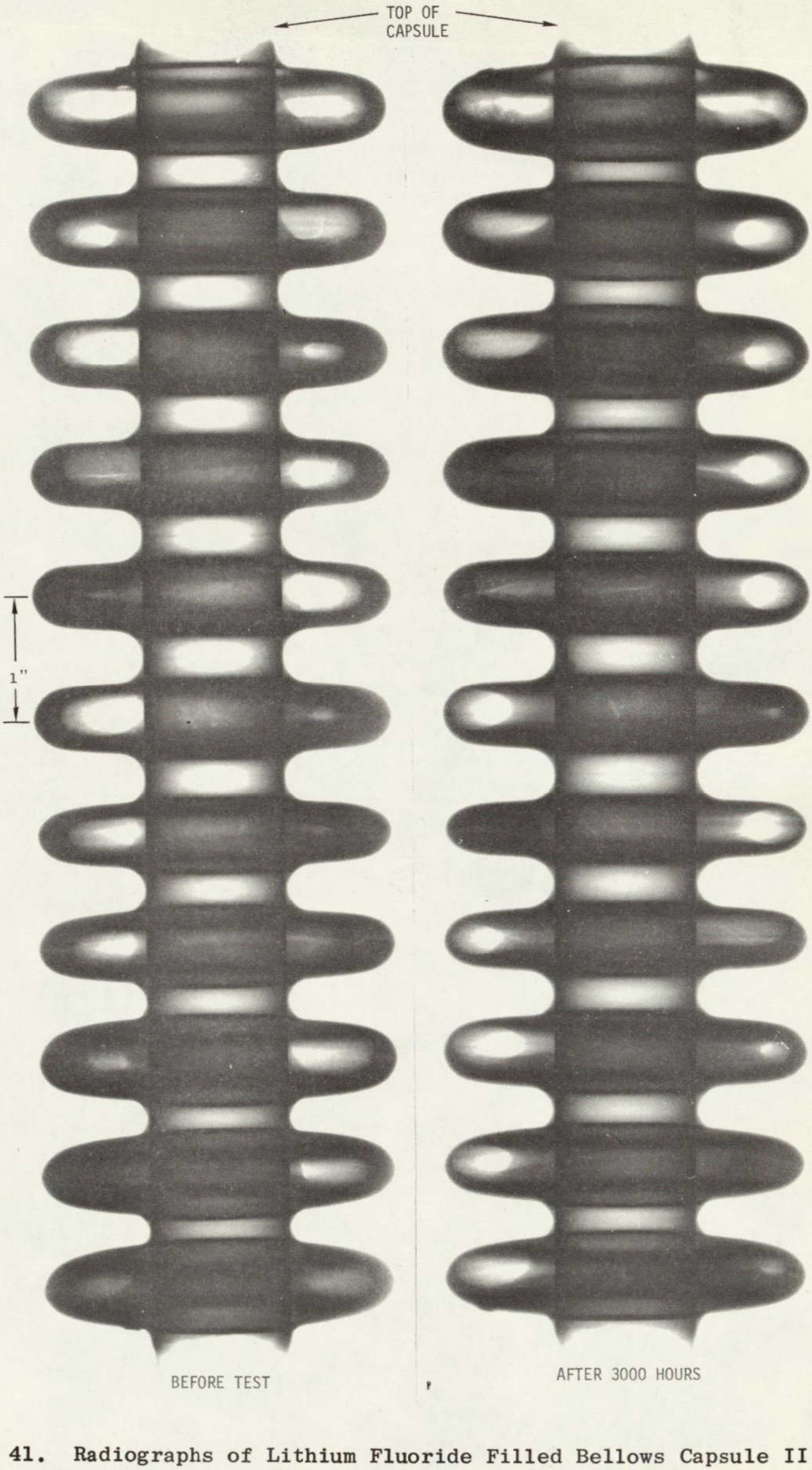
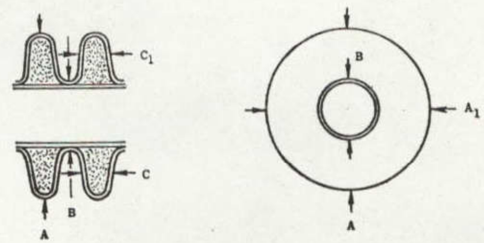


Figure 41. Radiographs of Lithium Fluoride Filled Bellows Capsule II Before and After 3000 Hours of Testing. White Areas are Voids in the Lithium Fluoride.

TABLE IX. LITHIUM FLUORIDE BELLOWS CAPSULE II MEASUREMENTS BEFORE TEST, AFTER 1000 HOURS OF TESTING, AND AFTER 3000 HOURS OF TESTING.



Convolution Number	Location	A Before Test	A After 1000 Hrs	A After 3000 Hrs	A_1 Before Test	A_1 After 1000 Hrs	A_1 After 3000 Hrs	B Before Test	B After 1000 Hrs	B After 3000 Hrs	C Before Test	C After 1000 Hrs	C After 3000 Hrs	C_1 Before Test	C_1 After 1000 Hrs	C_1 After 3000 Hrs
1	Capsule Bottom	3.261	3.252	3.259	3.254	3.259	3.259	1.33	1.33	1.33	0.57	0.56	0.57	0.56	0.57	0.57
2		3.261	3.263	3.265	3.283	3.285	3.288	1.35	1.35	1.34	0.54	0.57	0.56	0.54	0.58	0.55
3		3.290	3.287	3.290	3.275	3.277	3.280	1.34	1.34	1.34	0.57	0.61	0.61	0.58	0.62	0.60
4		3.283	3.281	3.286	3.276	3.280	3.284	1.34	1.34	1.34	0.57	0.58	0.58	0.58	0.59	0.58
5		3.264	3.269	3.265	3.265	3.265	3.267	1.33	1.34	1.34	0.55	0.58	0.54	0.56	0.56	0.56
6		3.299	3.305	3.303	3.297	3.301	3.305	1.33	1.34	1.34	0.65	0.65	0.65	0.64	0.66	0.65
7		3.304	3.311	3.312	3.306	3.304	3.310	1.33	1.34	1.34	0.65	0.67	0.66	0.66	0.66	0.66
8		3.309	3.310	3.312	3.307	3.305	3.312	1.33	1.34	1.34	0.65	0.67	0.66	0.65	0.67	0.66
9		3.309	3.314	3.313	3.310	3.309	3.314	1.33	1.34	1.34	0.67	0.68	0.67	0.66	0.68	0.68
10		3.310	3.311	3.315	3.309	3.306	3.313	1.33	1.33	1.34	0.65	0.66	0.66	0.66	0.66	0.65
11	Capsule Top	3.293	3.294	3.293	3.302	3.302	3.302	1.33	1.34	1.34	0.64	0.65	0.65	0.65	0.65	0.65

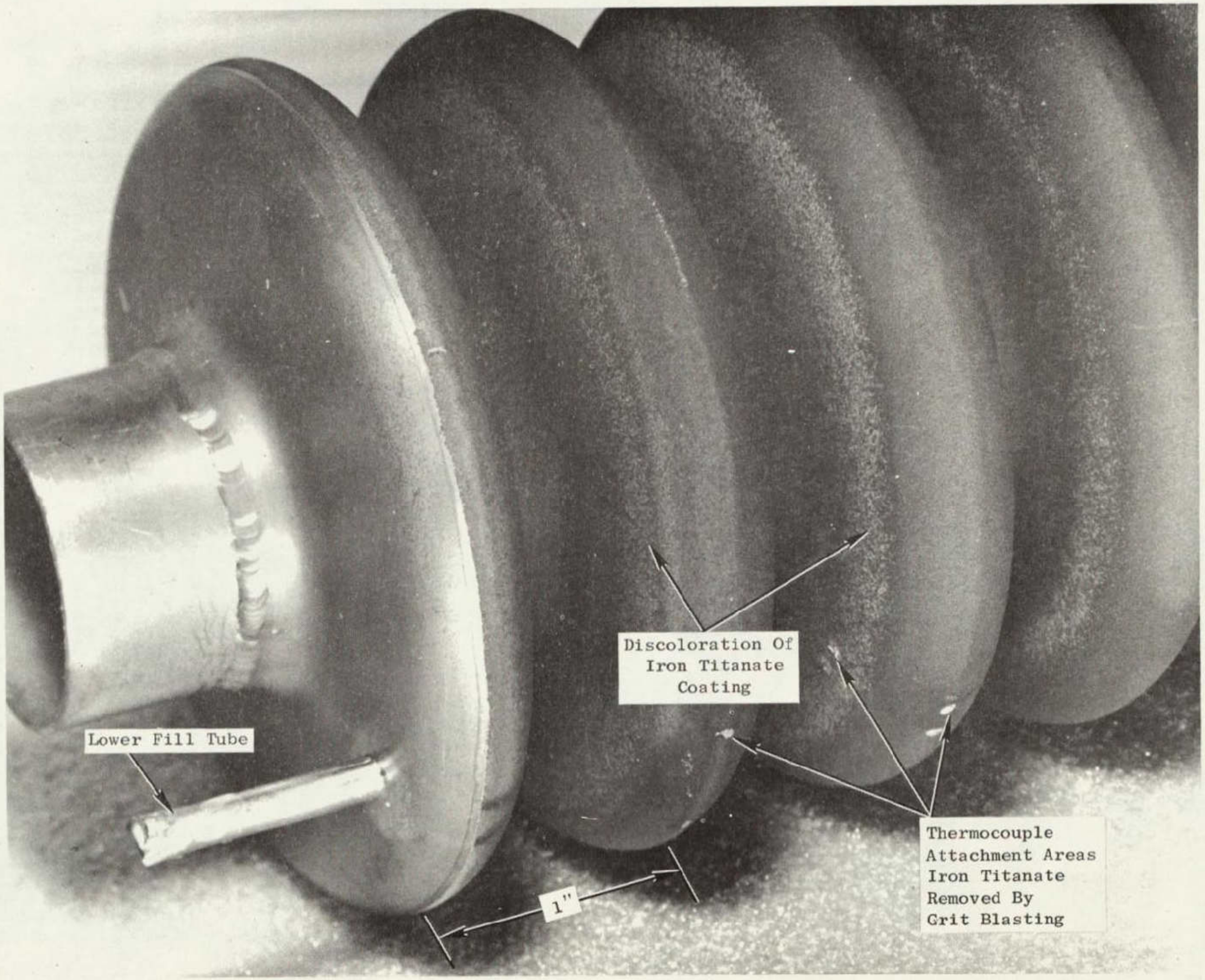


Figure 42. Bellows Capsule II After 3000 Hours of Test Showing Discoloration of the Iron-Titanate Coating. (CDC10984)

3. Bellows Capsule III

The test setup for Bellows Capsule III was primarily the same as for Bellows Capsule II. Bellows Capsule III was filled with lithium fluoride and the fill tubes sealed using the same procedure as employed in Bellows Capsule II. Bellows Capsule II leaked at the lower fill tube after 3000 hours of testing, and subsequent examination indicated the leak resulted from improper sealing techniques. Although the sealing procedures were modified, after Bellows Capsule III had been filled resealing of Bellows Capsule III using the improved techniques was deemed impractical. As a result, a molybdenum heat sink was installed around the lower fill tube of Bellows Capsule III to maintain the lithium fluoride in this tube in the frozen state throughout the test and thereby reduce the possibility of leakage. The heat sink, shown installed on the capsule in Figure 43, is a 0.500-inch diameter split molybdenum rod which has a hole in it the same size as the fill tube and fits tightly around it. The rod is of sufficient length to extend below the base plate of the capsule stand and is not insulated in this area to enhance heat losses by radiation to the cold chamber walls.

A second lithium fluoride leak detector was installed in the capsule test facility. As a reserve in case the first detector should become inoperative during testing and thereby avoid a shutdown of the test.

The Lithium Fluoride Bellows Capsule III Test Facility is shown in Figure 44 prior to installation of heater and heat shielding. The same pumpdown and bakeout procedure used for Bellows Capsules I and II was used for Bellows Capsule III. The vacuum pressure recorded at the completion of the bakeout of the vacuum chamber was 1×10^{-8} torr.

a. Steady State Measurements

During initial testing, thermal measurements were made of Bellows Capsule III to establish radiation losses during the cooling cycle. Steady state temperature measurements were performed at temperatures between 1080°F and 1680°F with no air flowing through the center cooling tube and with 19 scfm of air flowing through the cooling tube. The temperature profiles in the capsule, measured with a Leeds and Northrup Potentiometer at each steady state condition, are presented in Table X.

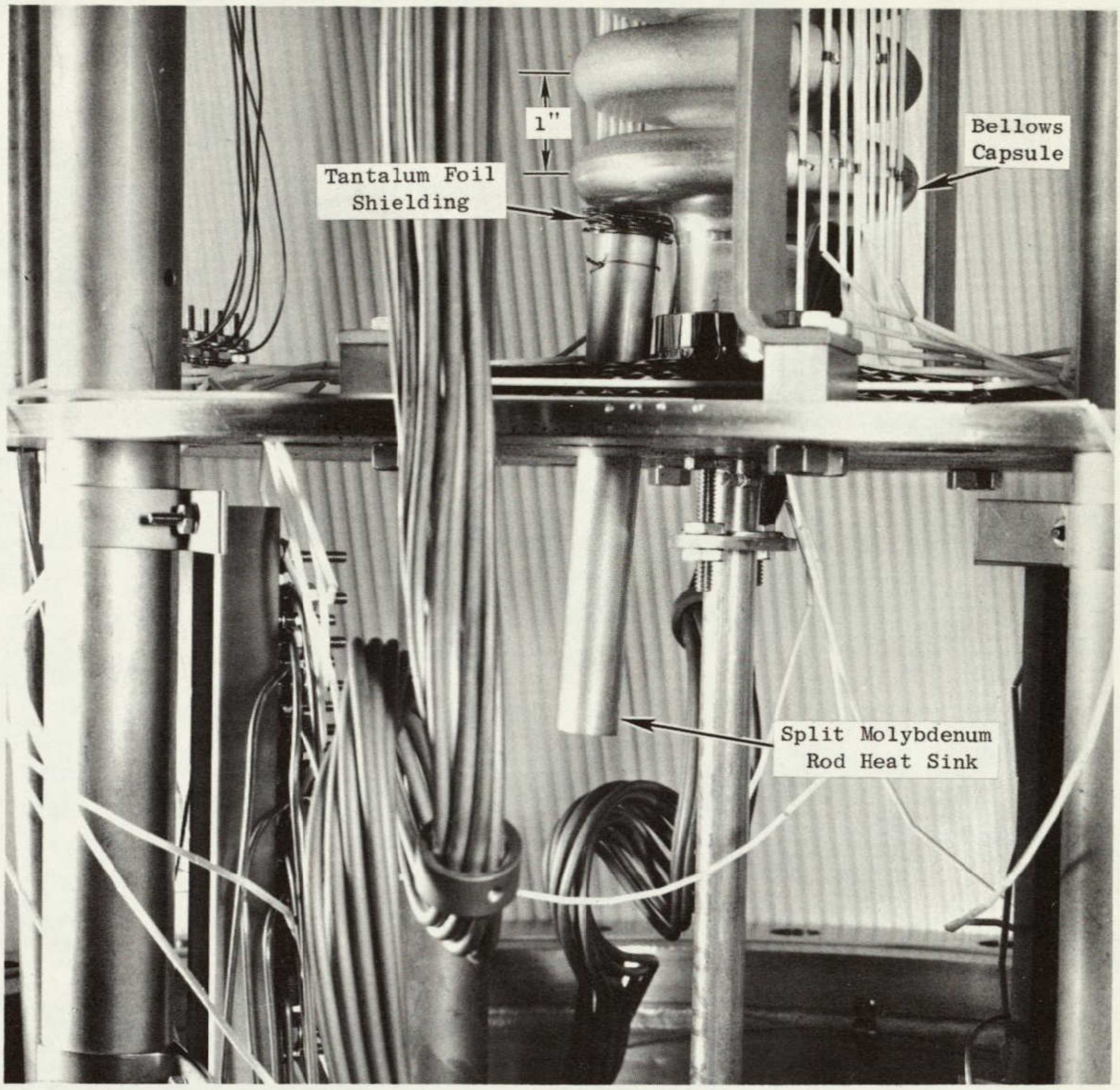


Figure 43. Lower Portion of the Lithium Fluoride Bellows Capsule III Test Facility Showing the Molybdenum Heat Sink Installed on the Fill Tube. (P69-1-27C)

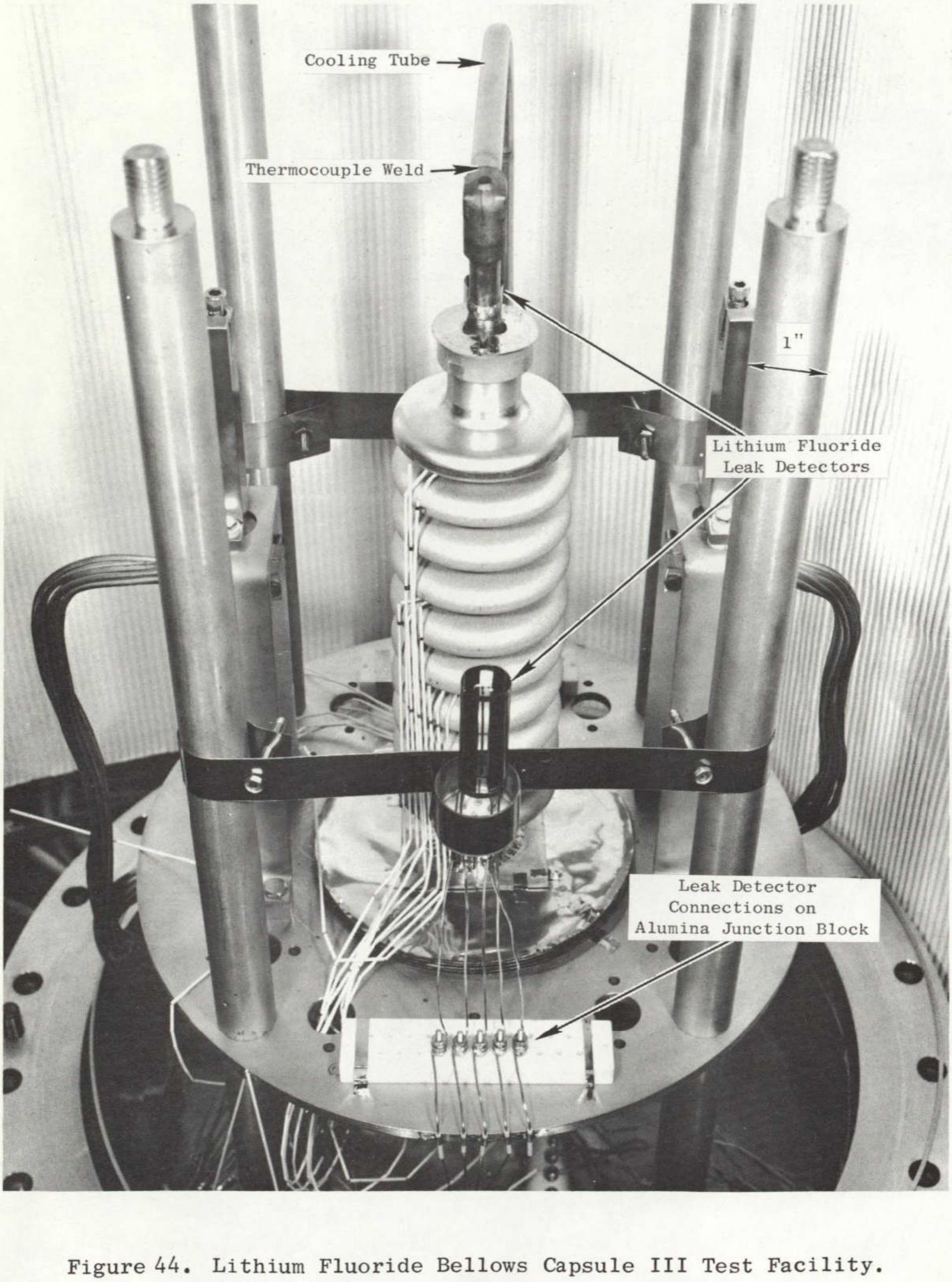
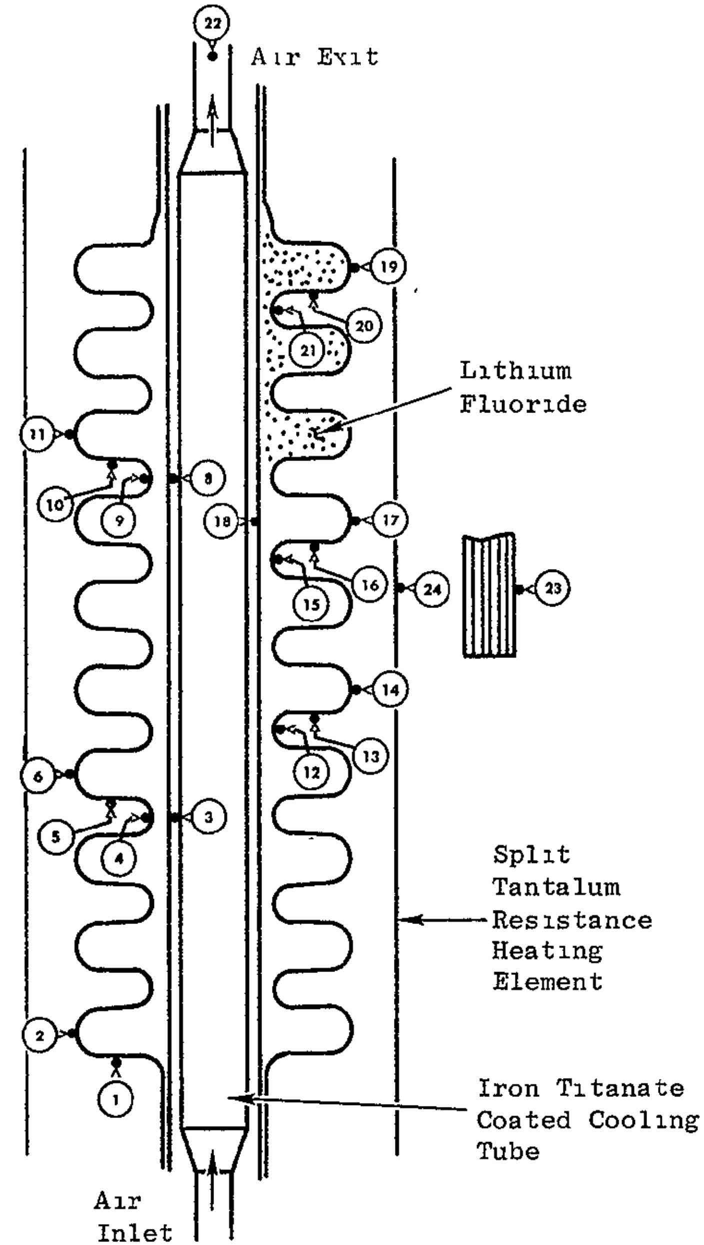


Figure 44. Lithium Fluoride Bellows Capsule III Test Facility. (P69-1-27A)

TABLE X. THE STEADY STATE TEMPERATURE PROFILES
LITHIUM FLUORIDE BELLOWS CAPSULE TEST III

	Temperature, °F				
Power, Watts	63	115	208	448	868
Air Flow SCFM	←No Air Flow→			19	19
Thermocouples					
1	1045	1341	1572	1380	1620
2	1045	1345	1575	1390	1628
3	1081	1377	1628	1301	1552
4	1082	1383	1625	1387	1632
5	1085	1391	1628	1408	1648
6	1088	1394	1633	1428	1665
7					
8	1079	1382	1618	1336	1608
9	1078	1383	1617	1370	1638
10	1077	1376	1613	1399	1652
11	1074	1383	1610	1405	1653
12	1086	--	1627	--	--
13	--	1386	--	1397	1642
14	1086	1390	1627	1422	1662
15	1082	1385	1622	1379	1646
16	1082	1348	1620	1392	--
17	--	1382	--	1414	1657
18	--	--	1615	--	--
19	1048	1382	1564	1358	1614
20	--	--	--	--	--
21	--	--	986	--	--
22	620	763	897	158	202
23	380	480	665	548	682
24	--	1410	--	1602	1869
Inlet Air	--	--	--	74	75



From the graph of the capsule temperatures* with air flowing in the cooling tube vs. the outer radiation shield temperature, shown in Figure 45, a shield temperature of 612°F can be found to correspond to a capsule temperature at the melting point of lithium fluoride. From the power temperature curve of the outer radiation shield, shown in Figure 46, a power value of 170 watts can be found to correspond to an outer shield temperature of 612°F. Neglecting small conduction losses, all the heat loss must occur by radiation when no air is flowing in the cooling tube. The power input, when the capsule is at steady state with no air flowing in the cooling tube, can therefore be equilibrated to radiation loss. Using Figures 45 and 46, it may be concluded that the radiative loss is equivalent to 170 watts when the capsule is at 1558°F. Application of 170 watts to the split tantalum heater during the cooling portion of the cycle compensates for this radiation loss. The thermocouples attached to the capsule heater indicate the heater temperature is approximately 10° higher than the capsule temperature during the cooling cycle with 170 watts applied to the split tantalum heater. Under this condition, capsule cooling occurs by heat rejection to the center cooling tube, a better simulation of the heat rejection mode in the proposed Solar Brayton Cycle Heat Receiver.

b. Test Initiation

Thermal cycling of Bellows Capsule III was initiated upon completing the steady state measurements.

The operating conditions of a typical cycle are shown in Table XI. The heat balance for the heating and cooling cycles is presented in Table XII. The heating cycle was set by increasing the power above that required to maintain the capsule at 1550°F by approximately 1750 Btu/hr. With this heat input, the lithium fluoride melted and reached a temperature of 1700°F in 51 minutes. The power was reduced at that time to 170 watts, that value previously determined to compensate for radiation losses (Figure 46). The cooling cycle under these conditions is 45 minutes in duration. The temperature profile of the center convolution for a typical cycle is shown in Figure 47.

*Thermocouple #6 (Table X) was selected to represent capsule temperature.

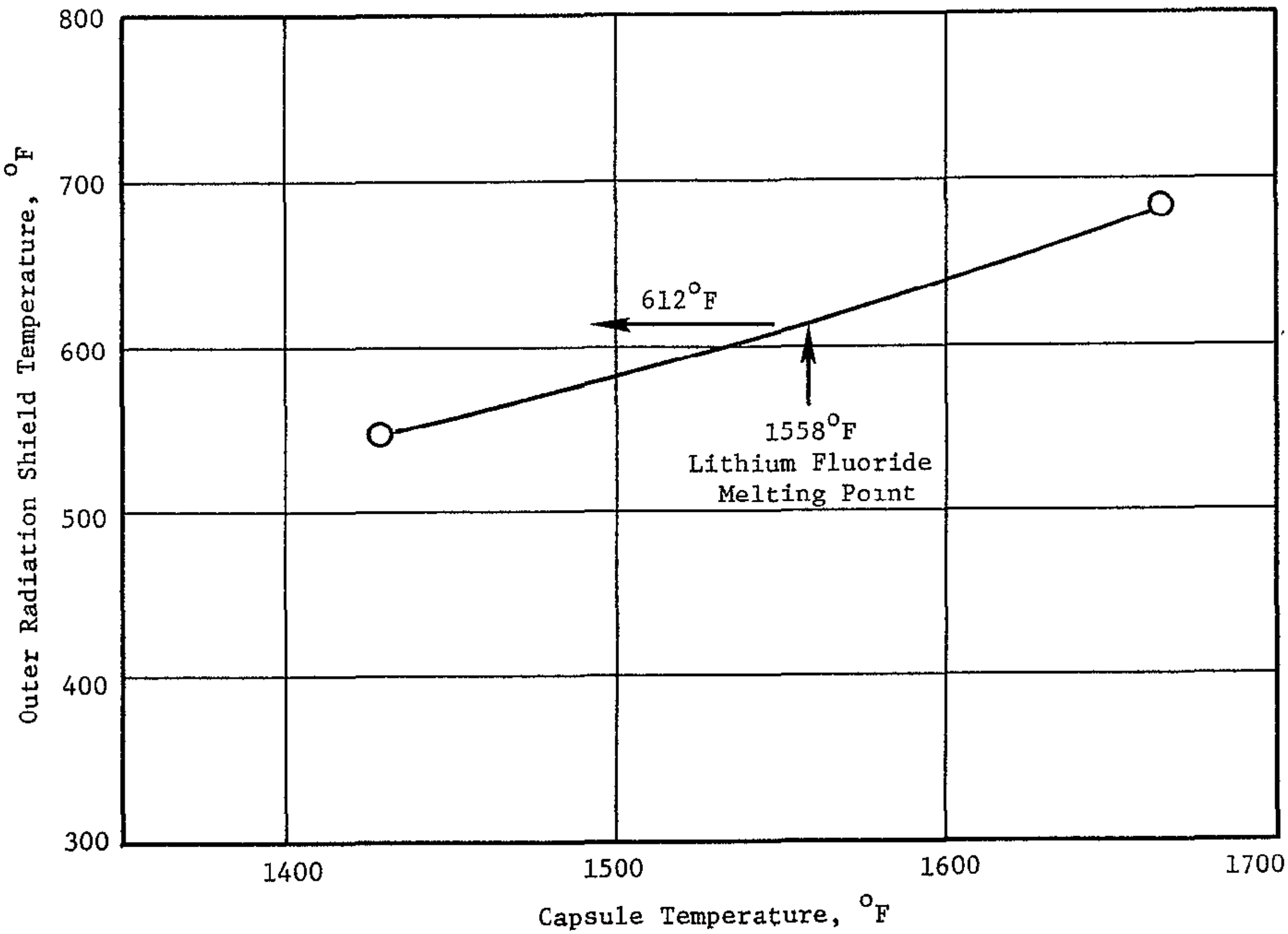


Figure 45. The Steady State Temperature Measurements of Lithium Fluoride Bellows Capsule Test III with 19 SCFM Air Flowing in the Cooling Tube.

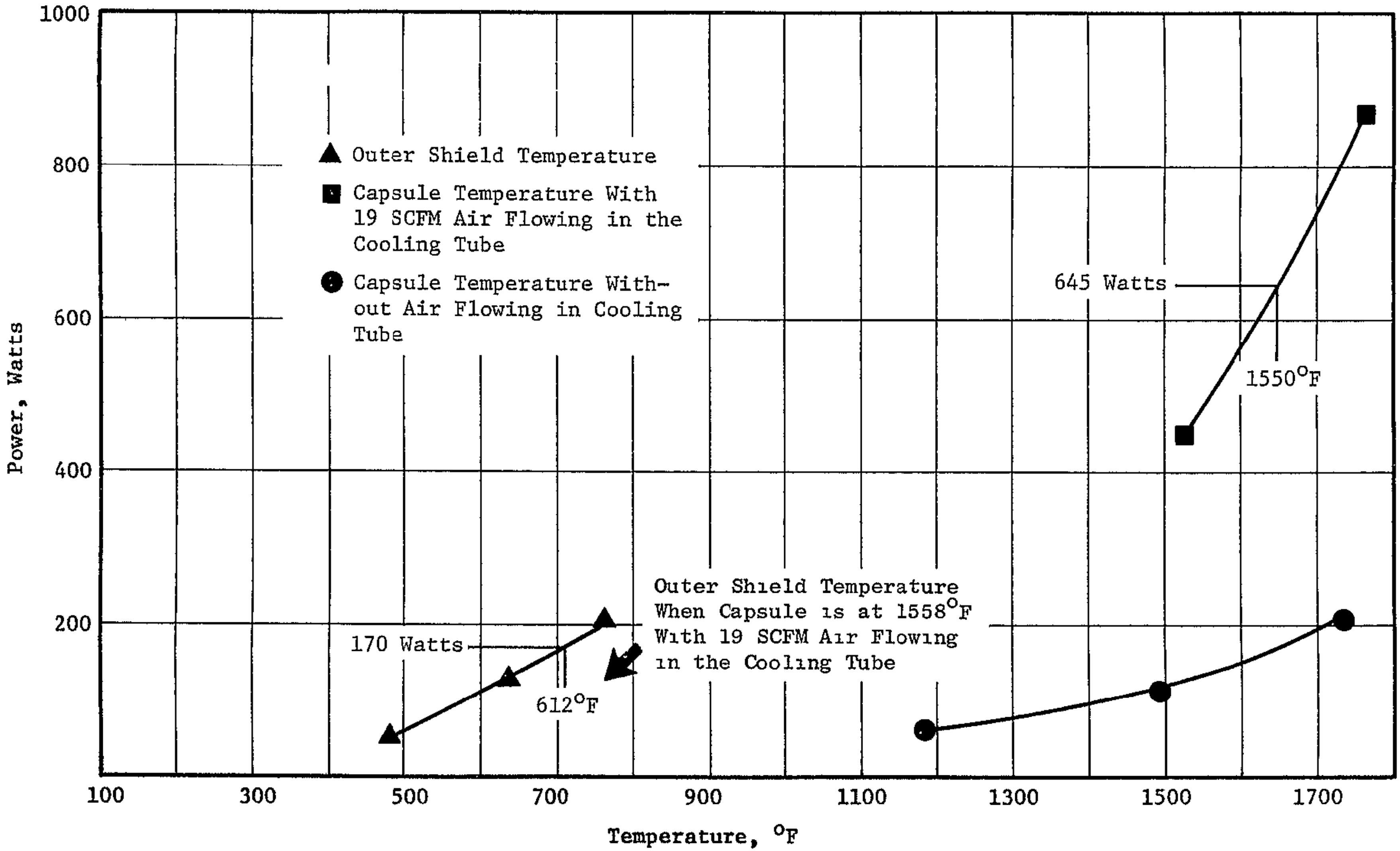


Figure 46. The Steady State Temperature Power Measurements of Lithium Fluoride Bellows Capsule III.

TABLE XI.

OPERATING CONDITIONS FOR THE LITHIUM FLUORIDE BELLOWS CAPSULE TEST III - 88th CYCLE

HEATING		
	Time	50 minutes
	Maximum Temperature*	1706°F
	Current	332 amps
	Voltage	4 volts
	Total Power	1328 watts
	Air Flow	19 scfm
	Chamber Pressure	3.4×10^{-8}
COOLING		
	Time	46 minutes
	Minimum Temperature*	1548°F
	Power	170 watts
	Air Flow	19 scfm
	Chamber Pressure	1.9×10^{-8}

* Measured with the same thermocouple.

TABLE XII.

HEAT BALANCE - LITHIUM FLUORIDE BELLOWS CAPSULE TEST III

Since:	Weight of Lithium Fluoride in Capsule	1118 gram
	Molecular Weight of Lithium Fluoride	25.94 g/moles
	Moles of Lithium Fluoride in Capsule	43.2 moles
	Heat of Solidification per Mole	25.7 Btu
	Total Heat of Solidification	1110 Btu

FUSION OF LITHIUM FLUORIDE

A.	Total Power	1328 watts
B.	Power Needed to Maintain 1550°F	645 watts
C.	Total Power to Melt Lithium Fluoride (A-B)	683 watts
D.	Radiation Loss at 1550°F	165 watts
E.	Power to Melt Lithium Fluoride (C-D)	518 watts
	....	(1750 Btu/hr)

Time Required to Melt the Lithium Fluoride

$$\frac{1110 \text{ Btu}}{1750 \text{ Btu/hr}} \quad x \quad 60 \text{ min/hr} \quad = \quad \underline{38 \text{ minutes}}$$

SOLIDIFICATION OF LITHIUM FLUORIDE

F.	Power to Compensate for Radiation Loss	170 watts
G.	Heat Dissipated by Cooling Tube at 1558°F with 19 scfm Air Flow and 120°F ΔT between Inlet and Exit Air	2500 Btu/hr

Time Required to Solidify the Lithium Fluoride

$$\frac{1110 \text{ Btu}}{2500 \text{ Btu/hr}} \quad x \quad 60 \text{ min/hr} \quad = \quad \underline{27 \text{ minutes}}$$

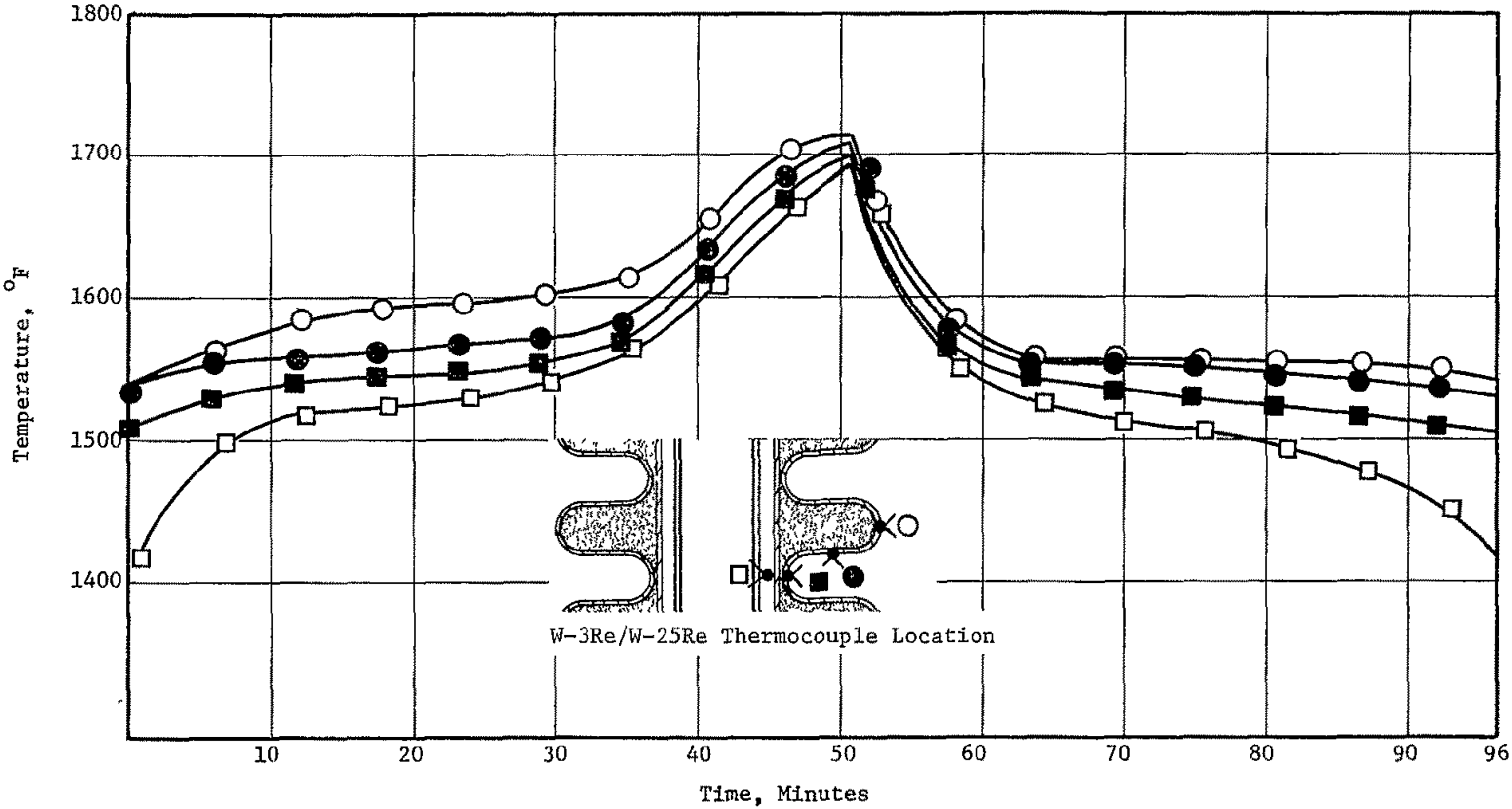


Figure 47. Temperature Profiles on the 88th Cycle of Lithium Fluoride Bellows Capsule Test III.

During the testing of Bellows Capsule III, the temperature data were plotted and examined at approximately 75-cycle intervals to denote any change which may be attributed to deformation of the capsule. No significant changes were noted.

On completion of the 5000 hours test time corresponding to the 3125th cycle (capsule temperature at 1545°F) the power to the split tantalum heater was kept at approximately 170 watts, the power required to compensate for radiation losses, until the capsule temperature reached 1450°F. At that time the capsule was shut off and allowed to cool to room temperature with air flowing in the cooling tube. This cooldown procedure was employed to simulate the cooldown in the Brayton Cycle Heat Receiver and thereby insure a similar freezing pattern.

Bellows Capsule III is shown in Figure 48 after removal from the test facility. Visual appearance of the grit blasted surface had not changed during the 5000 hours of testing.

Dimensional measurements taken of the capsule at that time are compared in Table XIII with those taken before test, after 1000 hours and after 3000 hours of testing. The data shows that dimensional changes, primarily of the center convolutions have occurred, especially the convolution thickness (Dimension C) and the distance between the convolutions (Dimension D).

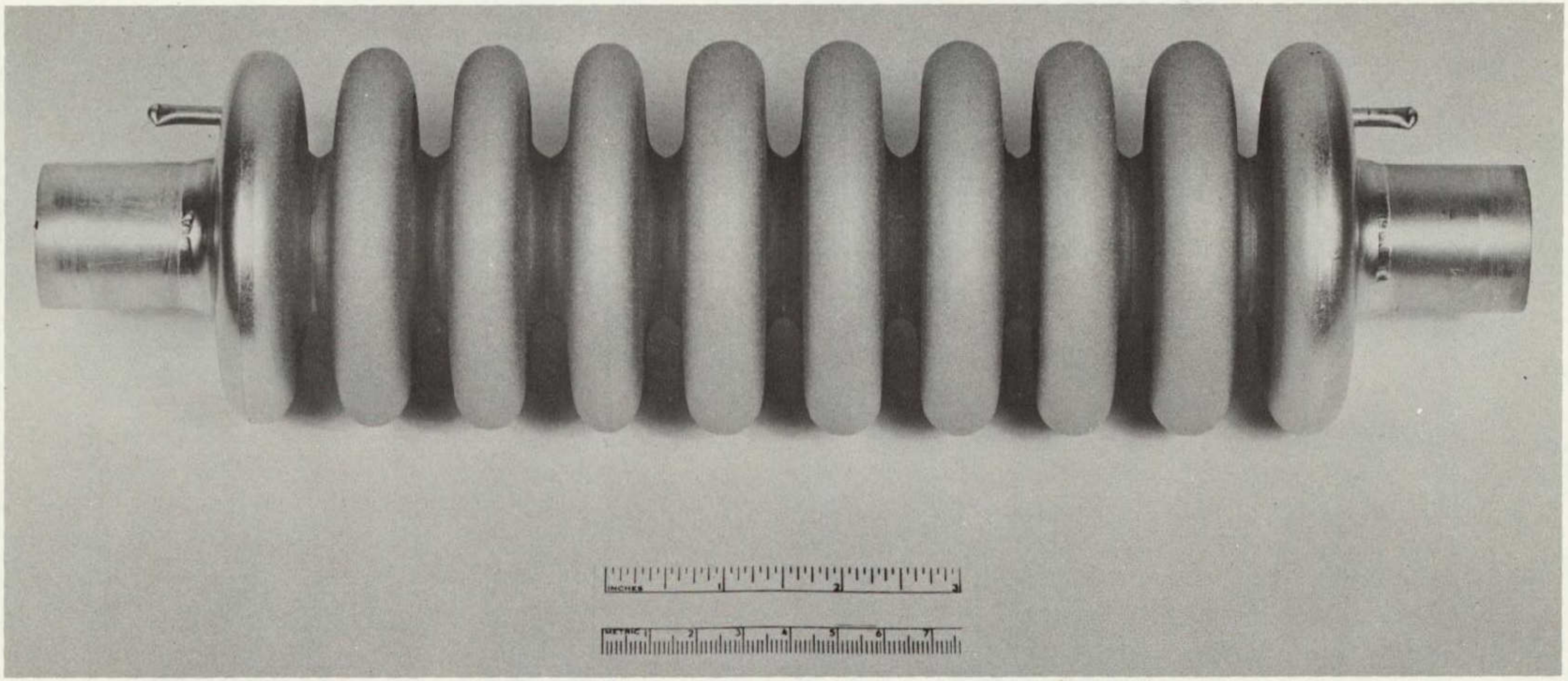


Figure 48. Lithium Fluoride Bellows Capsule III After 5000 Hours Test Time Corresponding to 3125 Thermal Cycles. (P69-10-13A)

TABLE XIII. LITHIUM FLUORIDE BELLOWS CAPSULE III MEASUREMENTS

Convolution Location		A Before Test	A After 1000 Hrs	A After 3000 Hrs	A After 5000 Hrs	A_1 (1) Before Test	A_1 (1) After 1000 Hrs	A_1 (1) After 3000 Hrs	A_1 (1) After 5000 Hrs	B Before Test	B After 1000 Hrs	B After 3000 Hrs	B After 5000 Hrs	B_1 (1) Before Test	B_1 (1) After 1000 Hrs	B_1 (1) After 3000 Hrs	B_1 (1) After 5000 Hrs
1 (Capsule top)	Measurement	3.139	3.138	3.138	3.139	3.127			3.128	1.341	1.350	1.341	1.341	1.338			1.337
	Change		-.001	-.001	.000				+.001		+.009	.000	.000				-.001
2	Measurement	3.187	3.184	3.185	3.185	3.186			3.186	1.338	1.338	1.337	1.338	1.340			1.341
	Change		-.003	-.002	-.002				.000		+.000	-.001	.000				+.001
3	Measurement	3.223	3.221	3.222	3.222	3.223			3.223	1.337	1.340	1.340	1.336	1.340			1.340
	Change		-.002	-.001	-.001				-.001		+.003	+.003	-.001				.000
4	Measurement	3.262	3.262	3.263	3.263	3.265			3.264	1.339	1.340	1.341	1.339	1.339			1.341
	Change		.000	+.001	+.001				-.001		+.001	+.002	.000				+.002
5	Measurement	3.309	3.308	3.308	3.309	3.308			3.308	1.345	1.350	1.344	1.346	1.350			1.347
	Change		-.001	-.001	.000				.000		+.005	-.001	+.001				-.003
6	Measurement	3.305	3.306	3.306	3.306	3.309			3.310	1.348	1.352	1.350	1.351	1.342			1.347
	Change		+.001	+.001	+.001				+.001		+.004	+.002	+.003				+.005
7	Measurement	3.311	3.310	3.311	3.311	3.309			3.311	1.344	1.345	1.348	1.346	1.347			1.350
	Change		-.001	.000	.000				+.002		+.001	+.004	+.002				+.003
8	Measurement	3.305	3.306	3.306	3.307	3.308			3.309	1.344	1.348	1.345	1.345	1.348			1.347
	Change		+.001	+.001	+.002				+.001		+.004	+.001	+.001				-.001
9	Measurement	3.307	3.308	3.308	3.307	3.311			3.307	1.350	1.349	1.349	1.351	1.348			1.345
	Change		+.001	+.001	.000				-.004		-.001	-.001	+.001				-.003
10	Measurement	3.291	3.291	3.292	3.291	3.297			3.299								
	Change		.000	+.001	.000				-.002								

(1) Dimensions A_1 and B_1 were inaccessable with capsule in place in test facility and were not taken after 1000 hours and 3000 hours.

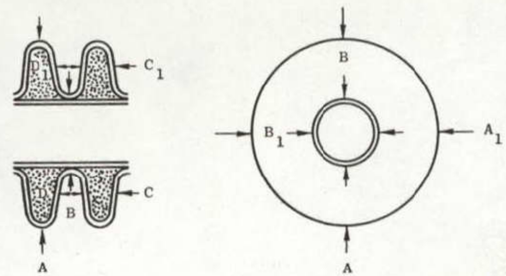


TABLE XIII. LITHIUM FLUORIDE BELLOWS CAPSULE III MEASUREMENTS (Cont)

Convolution Location		C Before Test	C After 1000 Hrs	C After 3000 Hrs	C After 5000 Hrs	C_1 Before Test	C_1 After 1000 Hrs	C_1 After 3000 Hrs	C_1 After 5000 Hrs
1 (Capsule top)	Measurement	0.616	0.616	0.619	0.617	0.621	0.620	0.623	0.622
	Change		.000	+.003	+.001		-.001	+.002	+.001
2	Measurement	0.623	0.623	0.624	0.625	0.626	0.627	0.631	0.627
	Change		.000	+.001	+.002		+.001	+.005	+.001
3	Measurement	0.620	0.619	0.619	0.619	0.620	0.621	0.624	0.625
	Change		-.001	-.001	-.001		+.001	+.004	+.005
4	Measurement	0.620	0.621	0.619	0.619	0.617	0.618	0.616	0.617
	Change		+.001	-.001	-.001		+.001	-.001	.000
5	Measurement	0.685	0.686	0.679	0.677	0.689	0.689	0.685	0.681
	Change		+.001	-.006	-.008		.000	-.004	-.008
6	Measurement	0.685	0.686	0.678	0.675	0.684	0.683	0.676	0.674
	Change		+.001	-.007	-.010		-.001	-.008	-.010
7	Measurement	0.681	0.680	0.669	0.660	0.684	0.682	0.680	0.673
	Change		-.001	-.012	-.020		-.002	-.004	-.011
8	Measurement	0.679	0.677	0.668	0.648	0.681	0.677	0.671	0.650
	Change		-.002	-.011	-.031		-.004	-.010	-.031
9	Measurement	0.686	0.685	0.685	0.683	0.683	0.683	0.688	0.685
	Change		-.001	-.001	-.003		.000	+.005	+.002
10	Measurement	0.656	0.663	0.660	0.655	0.662	0.661	0.663	0.661
	Change		+.007	+.004	-.001		-.001	+.001	-.001

Convolution Location		D Before Test	D After 1000 Hrs	D After 3000 Hrs	D After 5000 Hrs	D_1 Before Test	D_1 After 1000 Hrs	D_1 After 3000 Hrs	D_1 After 5000 Hrs
1 (Capsule top)	Measurement	0.350	0.350	0.350	0.351	0.353	0.353	0.353	0.357
	Change		.000	.000	+.001		.000	.000	+.004
2	Measurement	0.355	0.358	0.358	0.361	0.351	0.354	0.353	0.358
	Change		+.003	+.003	+.006		+.003	+.002	+.007
3	Measurement	0.361	0.365	0.362	0.366	0.368	0.371	0.370	0.378
	Change		+.004	+.001	+.005		+.003	+.002	+.010
4	Measurement	0.346	0.349	0.356	0.358	0.346	0.349	0.350	0.356
	Change		+.003	+.010	+.012		+.003	+.004	+.010
5	Measurement	0.304	0.305	0.314	0.317	0.309	0.311	0.320	0.323
	Change		+.001	+.010	+.013		+.002	+.011	+.014
6	Measurement	0.310	0.310	0.325	0.335	0.303	0.304	0.311	0.317
	Change		.000	+.015	+.025		+.001	+.008	+.014
7	Measurement	0.305	0.307	0.314	0.312	0.308	0.308	0.317	0.316
	Change		+.002	+.009	+.007		.000	+.009	+.008
8	Measurement	0.309	0.308	0.310	0.315	0.299	0.300	0.298	0.307
	Change		-.001	+.001	+.006		+.001	-.001	+.008
9	Measurement	0.310	0.310	0.310	0.311	0.307	0.312	0.309	0.314
	Change		.000	.000	+.001		+.005	+.005	+.007

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III. EVALUATION

A. BELLOWS CAPSULE I

The evaluation of Bellows Capsule I consisted of visual examination, chemical analysis and cursory metallographic examination. It is believed the deformation observed in the bellows capsule, shown previously in Figure 29, occurred as a result of differential thermal expansion between the outer portion or apex of the convolutions and the inner portion or root of the convolutions. A schematic representation of this mechanism is depicted in Figure 49. Shortly after the heater power is applied, the lithium fluoride near the apex of the convolutions melts. At this time, the lithium fluoride in contact with the root of the convolutions and inner tube wall remains frozen and the wall temperatures in this location remain at 1550°F, 100°F below the temperature of the wall at the apex of the convolutions. The outer wall of the convolutions cannot expand freely as it is restrained by the lower temperature at the root of the convolutions. This thermal condition induces sufficient stress in the convolution wall to produce some plastic deformation as shown in Figure 49b. Free movement of the bellows is restrained at this point in the cycle by the solid lithium fluoride. Once all the lithium fluoride in the bellows convolutions is molten, some thermally induced stress can be attenuated by contraction of the bellows as shown in Figure 49c. The plastic deformation which occurs during each cycle producing the geometry shown in Figures 49b and 49c results in the observed deformation after the 625 cycles during the 1000 hour test represented by Figure 49f. The positive print of the radiograph negative of the capsule shown in Figure 50 indicates this deformation as well as the lithium fluoride freezing pattern. Less deformation occurred in the top and bottom convolutions as a result of their lower temperatures induced by end heat losses. The non-uniform distortion in the other convolutions possibly can be attributed to small differences in strength from convolution to convolution.

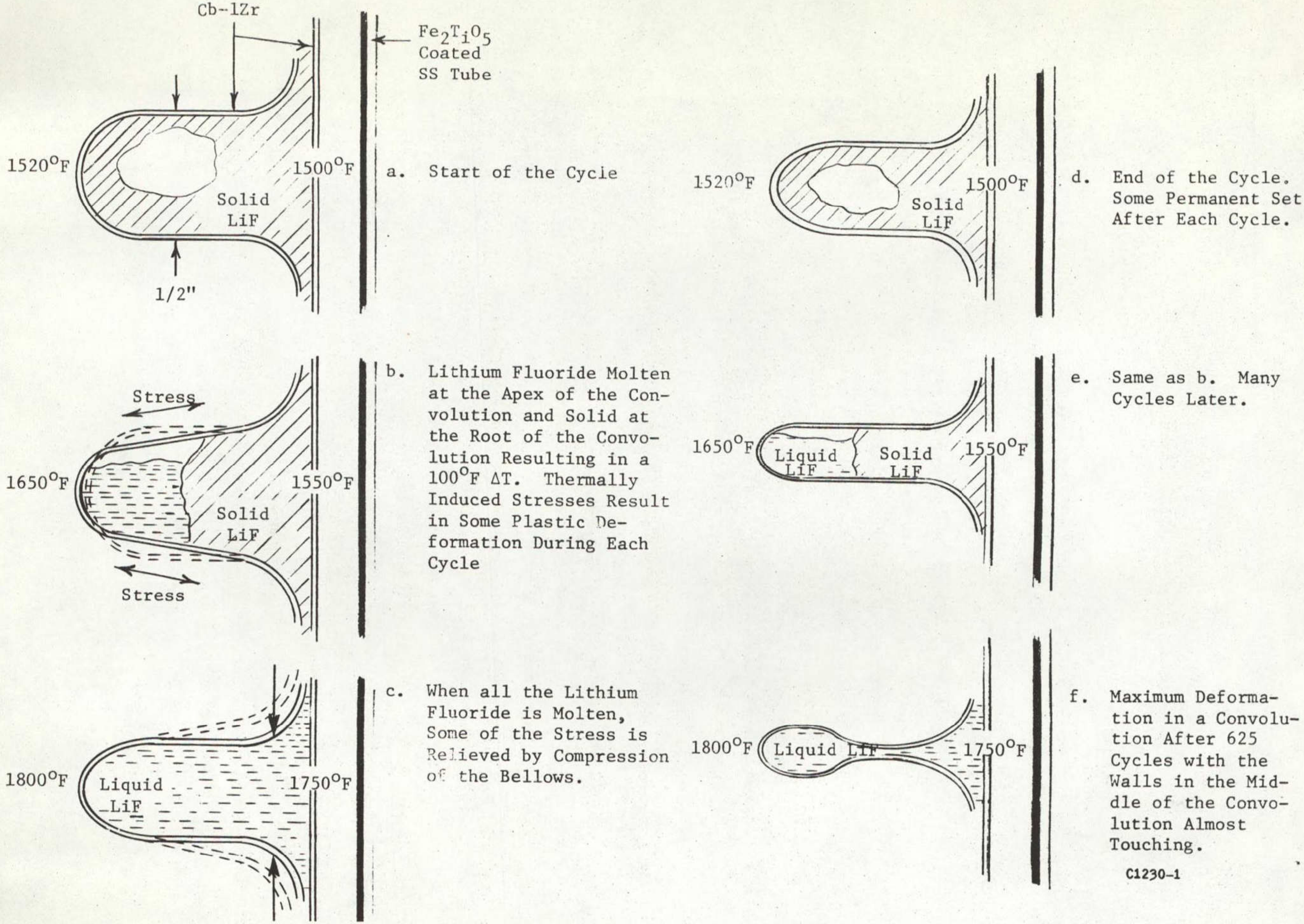

Figure 49. Schematic Indicating the Mechanism by which the Deformation in Bellows Capsule I Occurred.

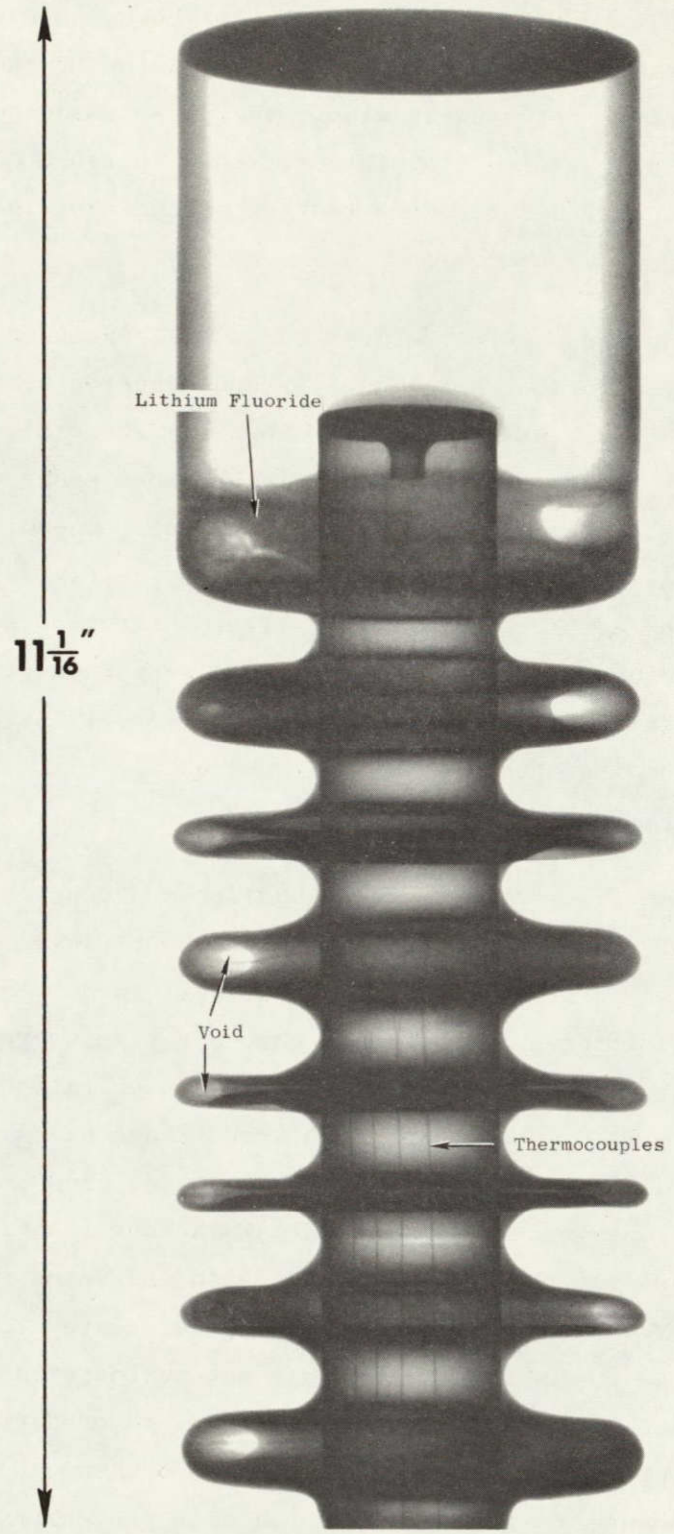

Figure 50. Radiograph of the Bellows Capsule I After 625 Cycles Between 1520°F and 1900°F (1000 Hour Test).

The capsule was cut in half longitudinally and the lithium fluoride was removed. Visual examination of capsule ID showed no evidence of corrosion. A section cut from the center convolution of the capsule was examined metallographically and it also revealed no evidence of corrosion. Chemical analysis of the same convolution, shown in Table XIV, indicated no contamination occurred during the testing of the capsule.

B. BELLOWS CAPSULE II

The posttest evaluation of Bellows Capsule II included chemical analysis, x-ray diffraction, metallography and electron microprobe analysis. The capsule was sectioned as shown in Figure 51, and was first separated into six sections by cutting between every other convolution. Each section was then halved by cutting perpendicular to section cuts. The inner tube was removed, and by gently tapping the bellows sections on a solid surface, the lithium fluoride was easily dislodged from the convolutions. The sections were then rinsed in hot water to remove any traces of lithium fluoride. Specimens were removed for evaluation from the areas shown in Figure 52.

1. Chemical Analysis

Specimens from the 3rd, 5th, 7th, and 11th convolutions were analyzed for oxygen, nitrogen, and hydrogen. The iron titanate coating was removed from all samples before analysis was performed. While very little changes in nitrogen and hydrogen concentrations were noted as a result of the test exposure, very high oxygen concentrations were found in the Cb-1Zr which had been coated with iron titanate. A schematic diagram of Bellows Capsule II is shown in Figure 53, and indicates the oxygen concentrations found in the Cb-1Zr at various locations. The maximum temperatures at those locations is also presented for comparison of the results. The higher the temperature, the greater the oxygen concentration. The top of the capsule was not coated with iron titanate. The oxygen concentration in the Cb-1Zr specimens removed from this area (171, 186 ppm) indicate very little increase in oxygen can be attributed to the vacuum exposure alone, when compared with the pretest values (178, 124, 140, 151 ppm).

TABLE XIV.

INTERSTITIAL ANALYSIS OF Cb-1Zr BELLOWS CAPSULE I[a]

Element[b]	Concentration, ppm Before Test	After Test
O	174,124[c]	181,160
N	25,15	27,36
H	5,2	1,2

(a) The capsule contained lithium fluoride and was cycled from 1520°F to 1900°F for 1000 hours.

(b) Analytical method - vacuum fusion

(c) Duplicate analysis

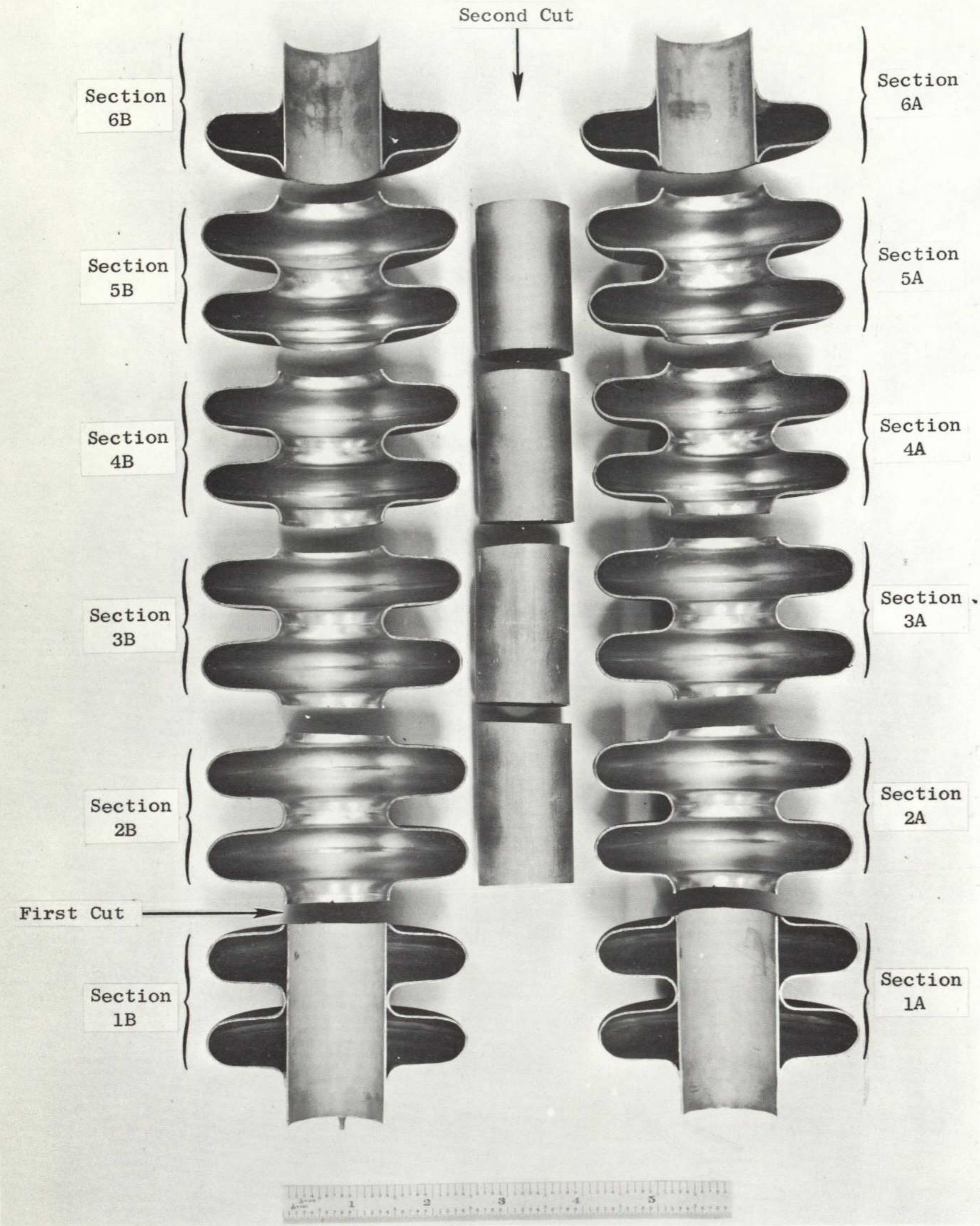

Figure 51. Sectioned Lithium Fluoride Bellows Capsule II After 3000 Hours of Testing and 1875 Thermal Cycles. (C68050714)

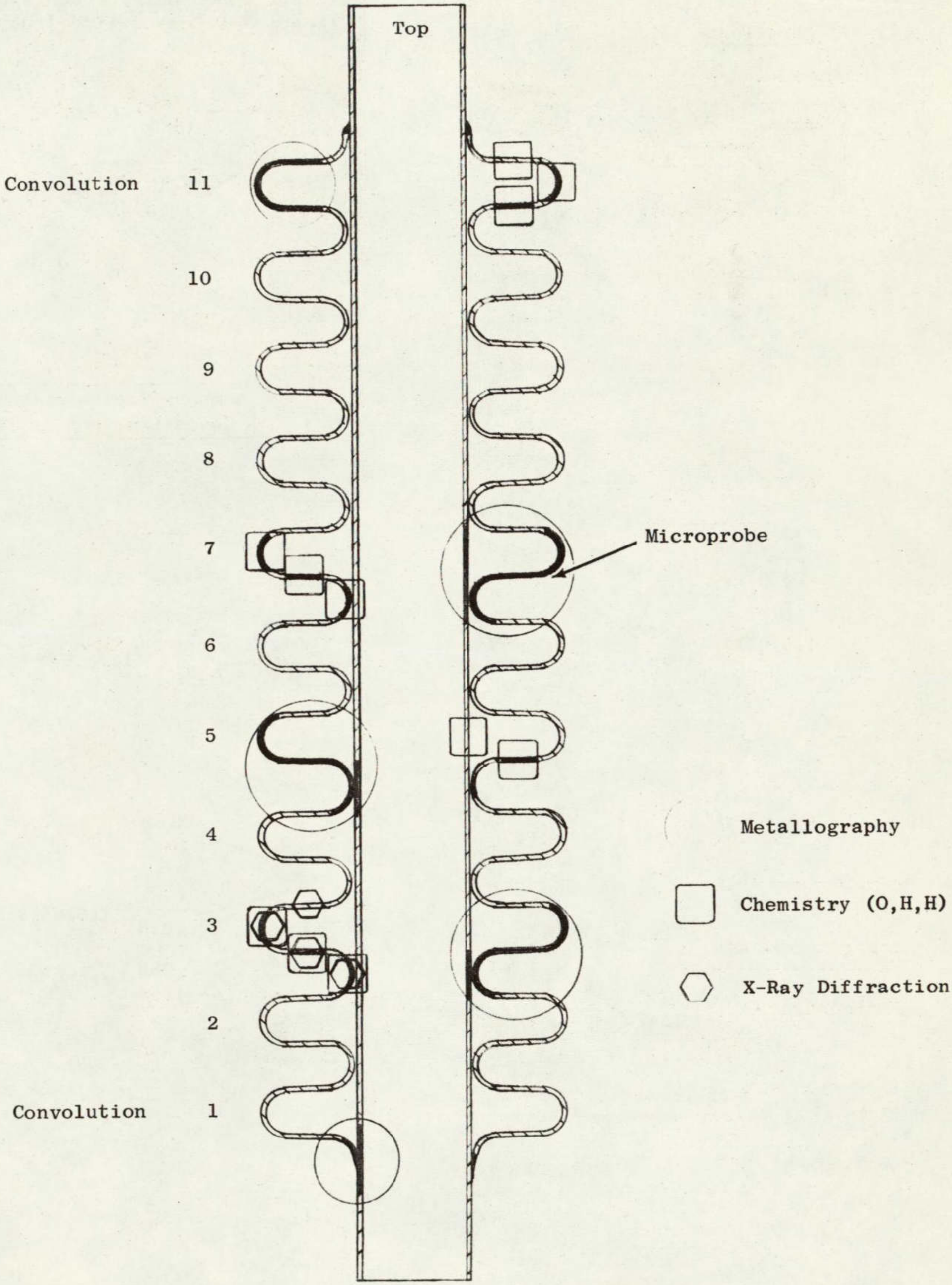

Figure 52. Location of Posttest Evaluation Specimens Taken for Bellows Capsule II.

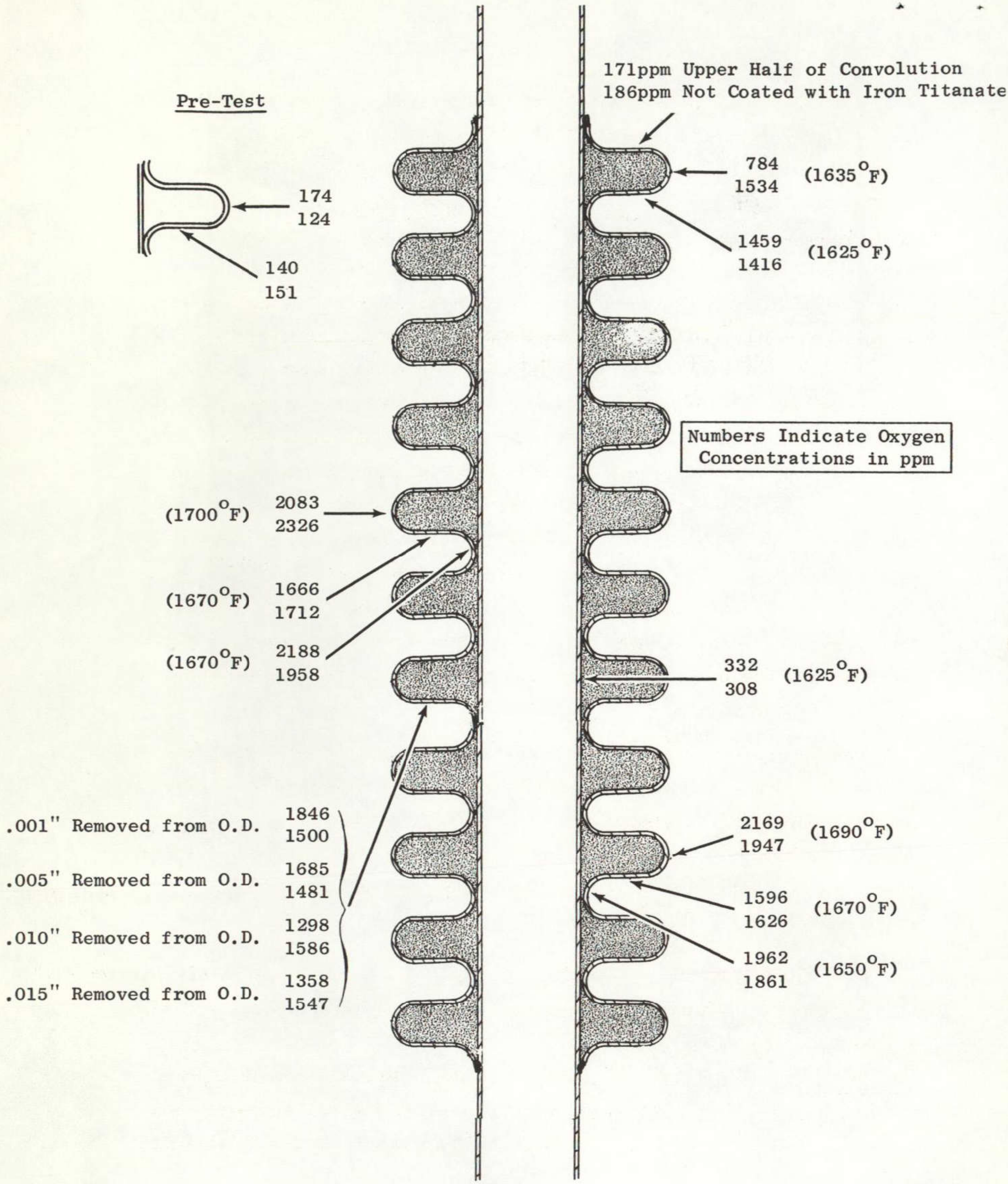

Figure 53. Oxygen Concentrations in the Cb-1Zr of Bellows Capsule II After 3000 Hours of Testing. The Capsule was Cycled Between 1500°F and 1700°F.

The relationship between capsule temperature, location of the sample, and oxygen concentration is summarized in Figure 54, which shows the oxygen concentration and temperature profile of Bellows Capsule II. Greater oxygen contamination was noted in the apex and root of the convolutions than in the midpoint areas, even though the temperature at the root of the convolutions was less than at the midpoint. Examination of the capsule after test revealed discoloration in the midpoint convolution areas. Closer examination during sectioning indicated the coating had deteriorated to such a degree in this area that the discoloration was actually the Cb-1Zr substrate showing through. Metallographic examination confirmed this observation. Examination of the before test specimens indicated the coating to be very thin in the midpoint of the convolutions as compared to the apex and root areas. This undoubtedly results from the manner in which the coating was applied to the capsule and its complex geometry. It is therefore reasonable that less contamination of the Cb-1Zr would occur in the midpoint convolution areas since less iron titanate was originally present, and the oxygen contamination results from the dissociation of the iron titanate.

The oxygen concentration gradient in the capsule wall was determined by gradient analysis. Four specimens cut from the midpoint of 5th convolution were machine ground from the coated surface to thicknesses of 0.029, 0.025, 0.020, and 0.015 inches analyzed. The oxygen concentrations, shown in Figure 53, indicate the oxygen was supplied at the coated surface.

2. Metallography

From metallographic examinations of Bellows Capsule II the following observations were made:

(1) The iron titanate coating had deteriorated, and in areas where the coating was thin (at the midpoint) approximately 50% of the Cb-1Zr was uncoated after 3000 hours of testing.

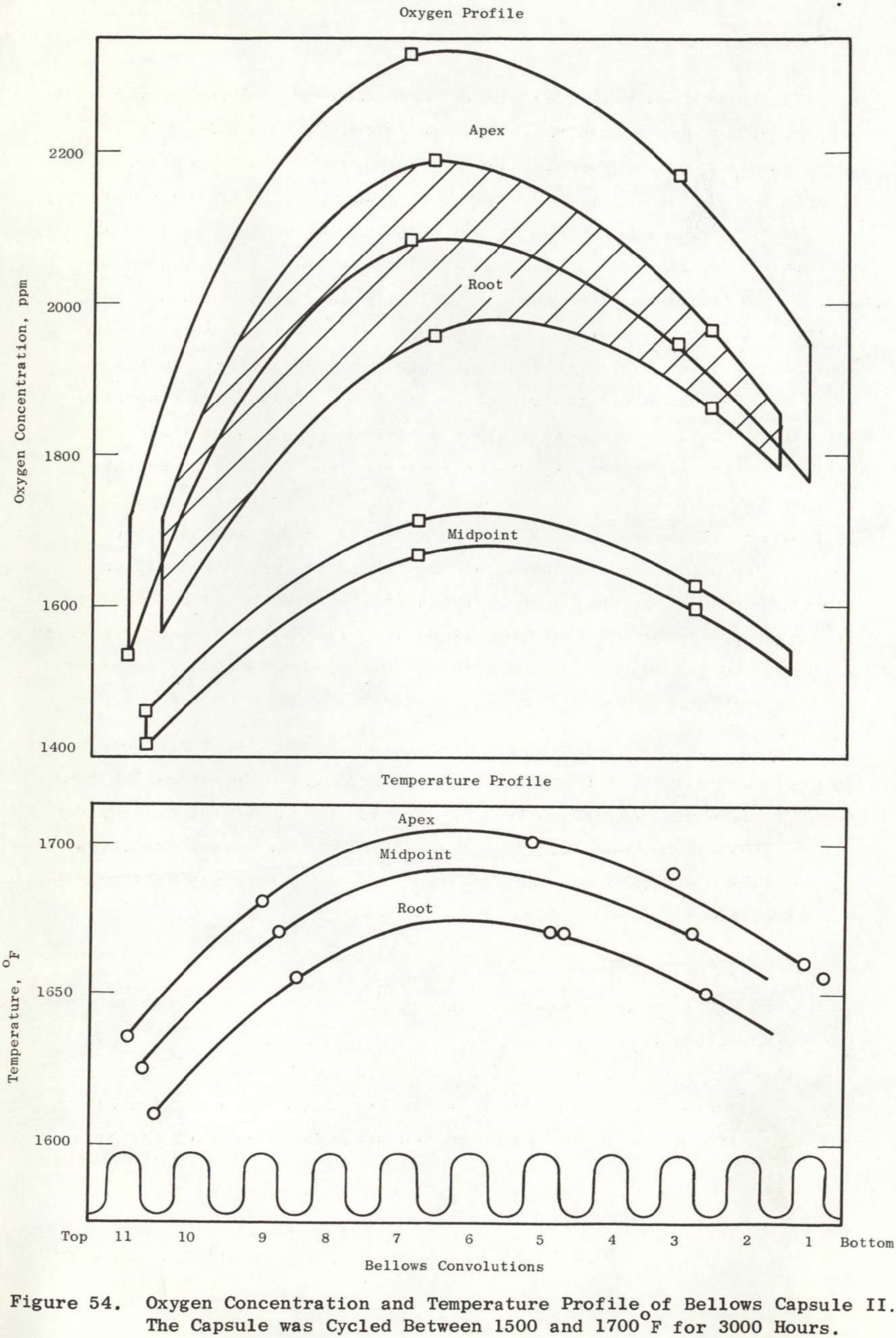

Figure 54. Oxygen Concentration and Temperature Profile of Bellows Capsule II. The Capsule was Cycled Between 1500 and 1700°F for 3000 Hours.

(2) A reaction had occurred between the iron titanate and the Cb-1Zr which resulted in the formation of a grain boundary phase that extended approximately 0.005 inch into the Cb-1Zr.

(3) No evidence of corrosion of the Cb-1Zr by the lithium fluoride was detected.

Photomicrographs of the iron titanate coating before and after test are shown in Figure 55. These photomicrographs show the coating to be considerably thicker at the apex and root areas than at the mid-point. The iron titanate coating underwent a definite change during the 3000 hours of testing. The coating decreased in thickness, and the thinly coated areas were only partially coated with iron titanate after test. A new phase, white in appearance, identified as iron by x-ray diffraction and microprobe analyses had also formed in the iron titanate coating.

Photomicrographs in Figure 56 are of specimens removed from areas of Bellows Capsule II which were at the highest temperature during testing. A grain boundary phase which extends to a depth of approximately 0.005 inch into the Cb-1Zr from the iron titanate coated side can be seen in several of the photomicrographs.

The anodic staining technique of identifying carbides, nitrides, oxides as reported by Crouse[(6)] was used in an effort to identify the grain boundary phase. After staining the specimen, the grain boundary phase shown in Figure 57 appeared yellow in color and similar in shade to columbium nitride identified in the referenced report.

Several hardness surveys, shown in Figures 58 and 59, were made on various sections of Bellows Capsule II. The measurements indicated maximum hardness values in the regions of the Cb-1Zr adjacent to the Cb-1Zr - iron titanate interface. A hardness gradient across the inner tube wall was also noted. Areas of the Cb-1Zr tube near the inside diameter of the tube, which faced the iron titanate coated stainless steel cooling tube across a 1/8-inch vacuum gap during testing, had the highest hardness values determined in the tube wall. The higher

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[(6)] Crouse, R. S., "Identification of Carbides, Nitrides and Oxides of Niobium and Niobium Alloys by Anodic Staining," ORNL-3821, July 1965.

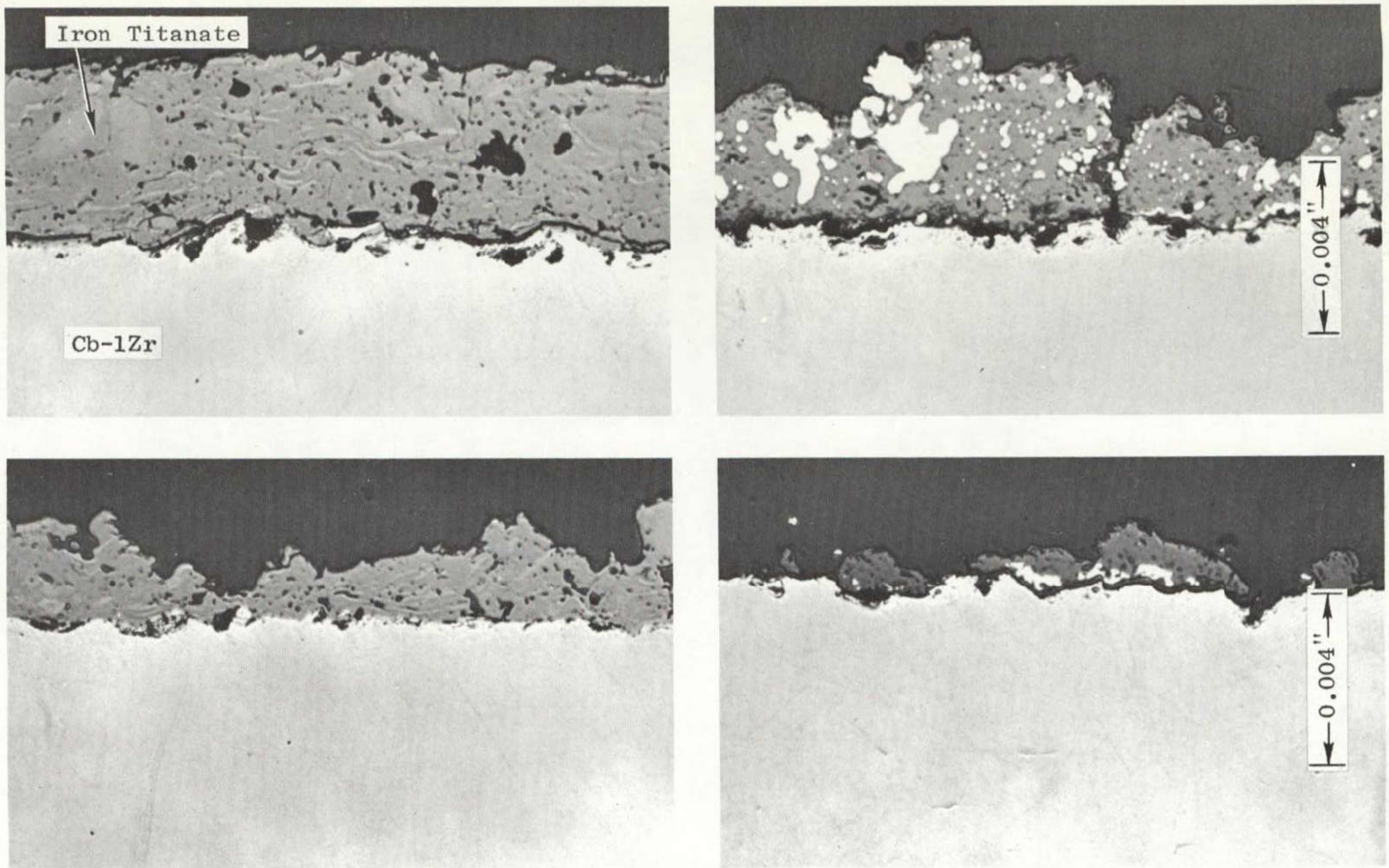


Figure 55. Typical Appearance of the Iron Titanate Coating on Lithium Fluoride Bellows Capsule II Before and After 3000 Hours of Testing. The Capsule was Cycled Between 1500 and 1700 °F. (F760112, F740415, F760111 & F740414)

As Polished Mag.: 250X

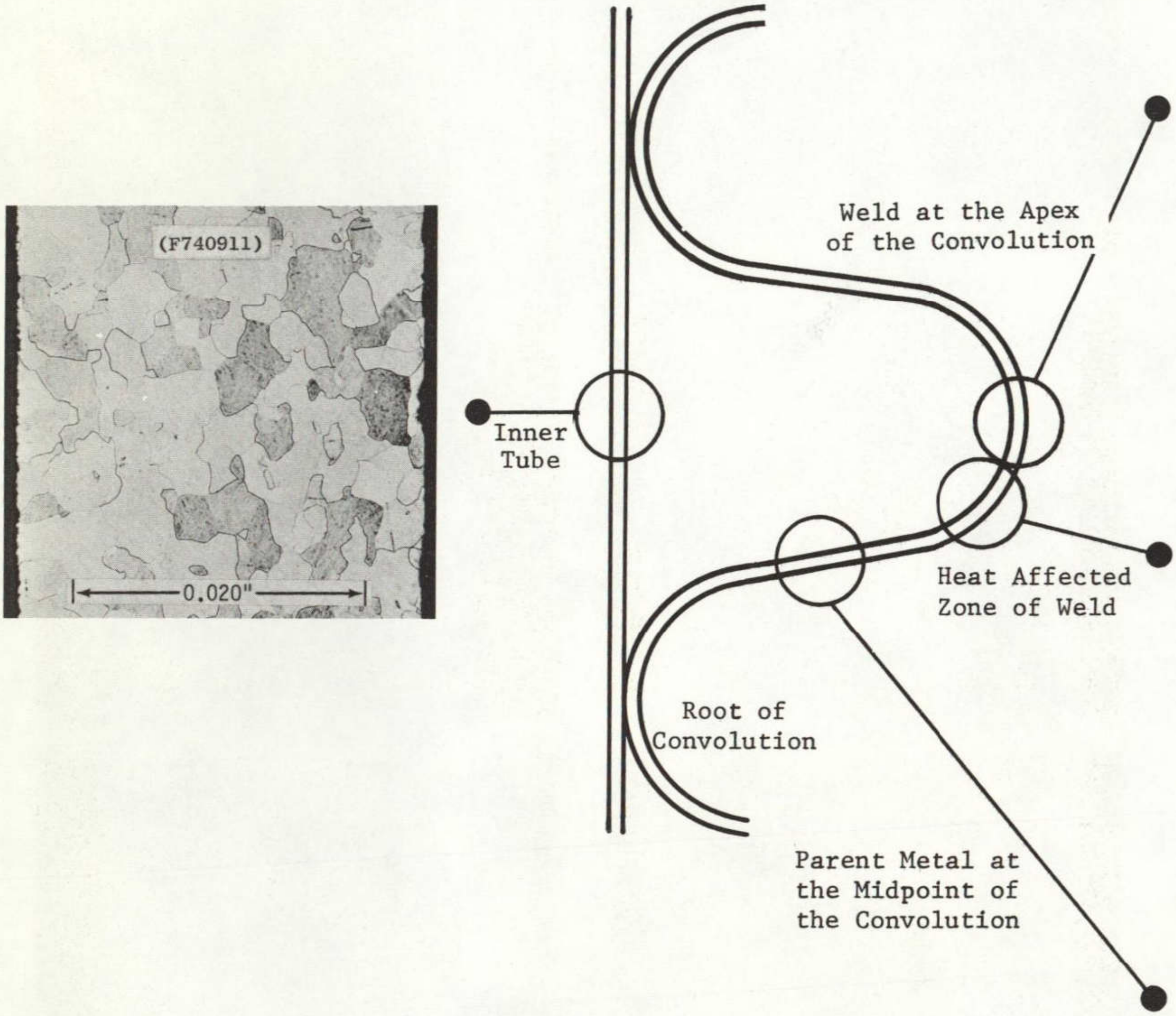


Figure 56. Photomicrographs of Lithium Fluoride Bellows Capsule II. The Cb-1Zr Capsule was Cycled Between 1500 and 1700^{o}F for 3000 Hours Corresponding to 1875 Thermal Cycles.

Etchant: 60 Glycerine, 20HNO_3, 20HF

After 3000 Hours of Testing | Before Test

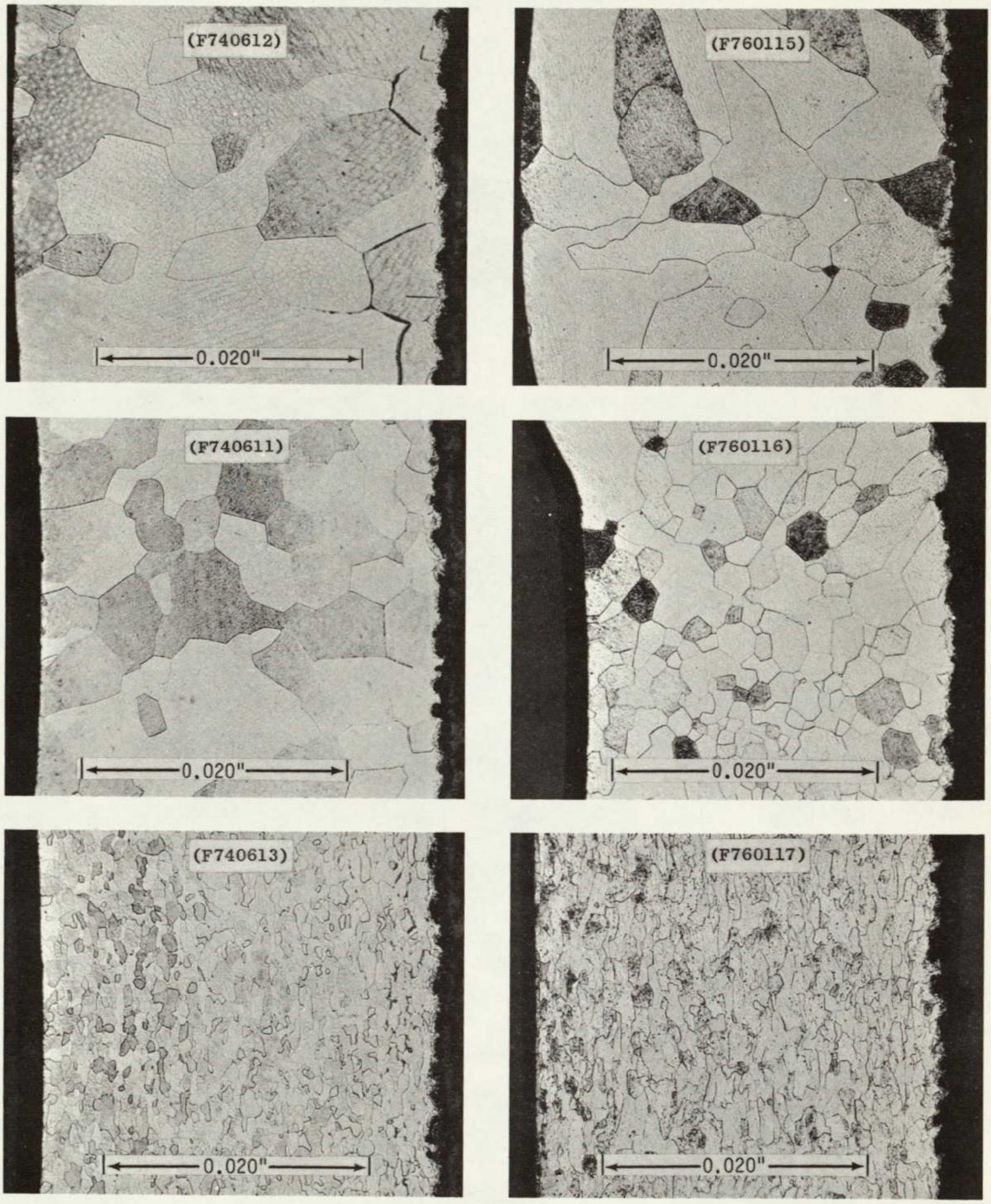
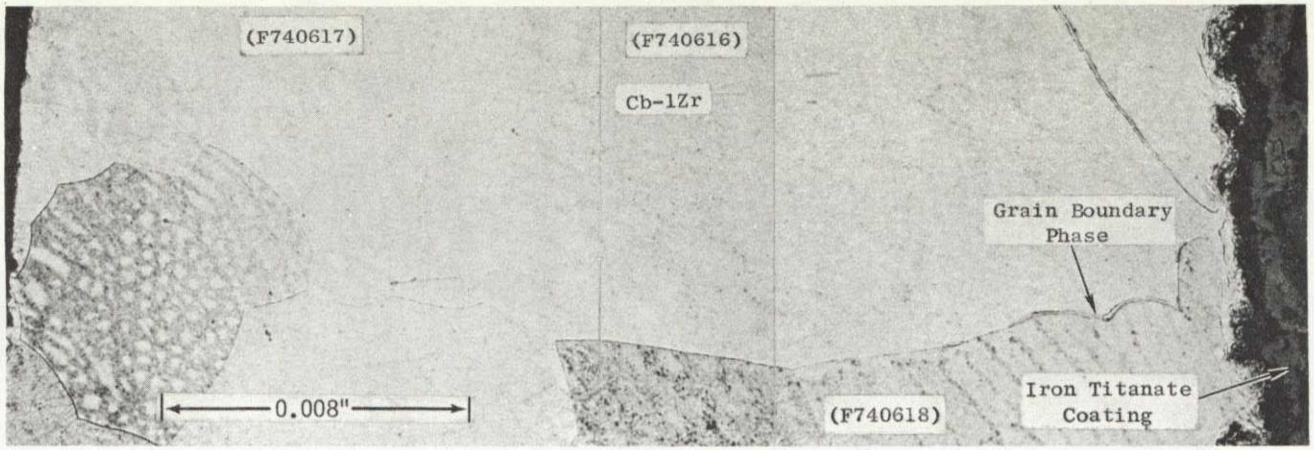


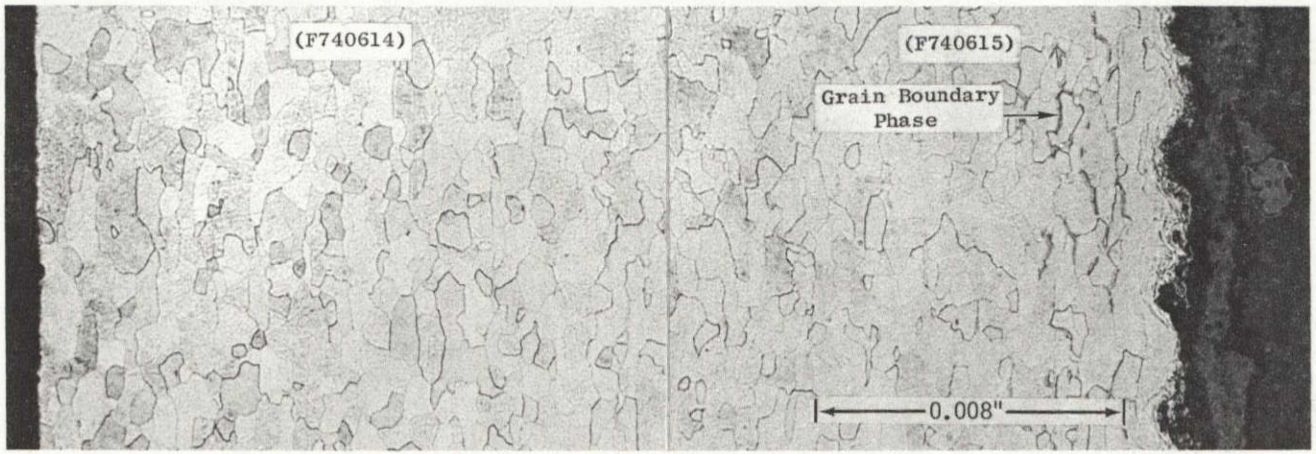
Figure 56 (Cont). Photomicrographs of Lithium Fluoride Bellows Capsule II. The Cb-1Zr Capsule was Cycled Between 1500 and 1700°F for 3000 Hours Corresponding to 1875 Thermal Cycles.

Etchant: 60 Glycerine, $20HNO_3$, 20HF

After 3000 Hours of Testing



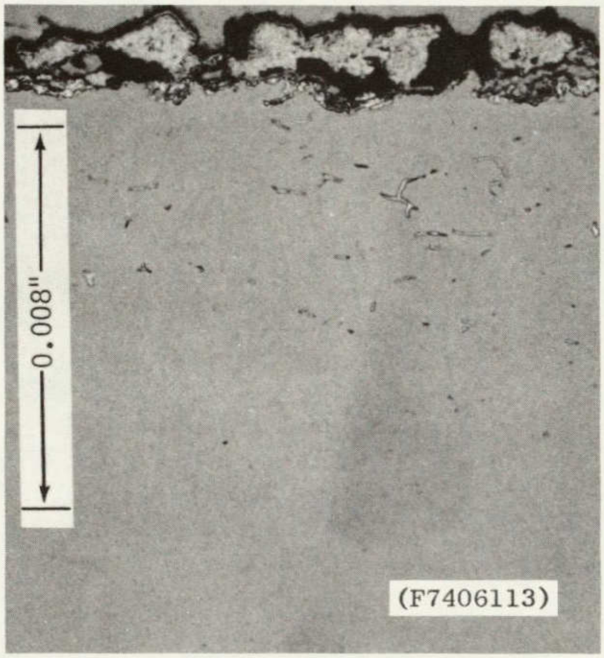
Weld at the Apex of the Convolution



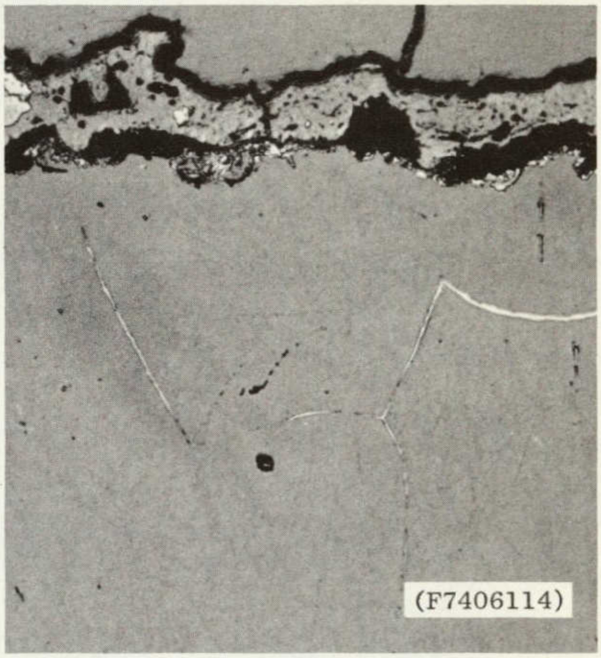
Parent Metal at the Midpoint of the Convolution

Figure 56 (Cont). Photomicrographs of Lithium Fluoride Bellows Capsule II. The Cb-1Zr Capsule was Cycled Between 1500 and 1700°F for 3000 Hours Corresponding to 1875 Thermal Cycles.

Etchant: 60 Glycerine, $20HNO_3$, 20HF



Midpoint of a
Convolution (Base Metal)



Apex of a
Convolution (Weld Nugget)

Figure 57. Grain Boundary Phase in Cb-1Zr Substrate of Iron Titanate Coated Bellows Capsule II Following 3000 Hours of Testing 1875 Thermal Cycles Between 1500 and 1700°F.

Electrolytic Stain Etch Mag.: 250X

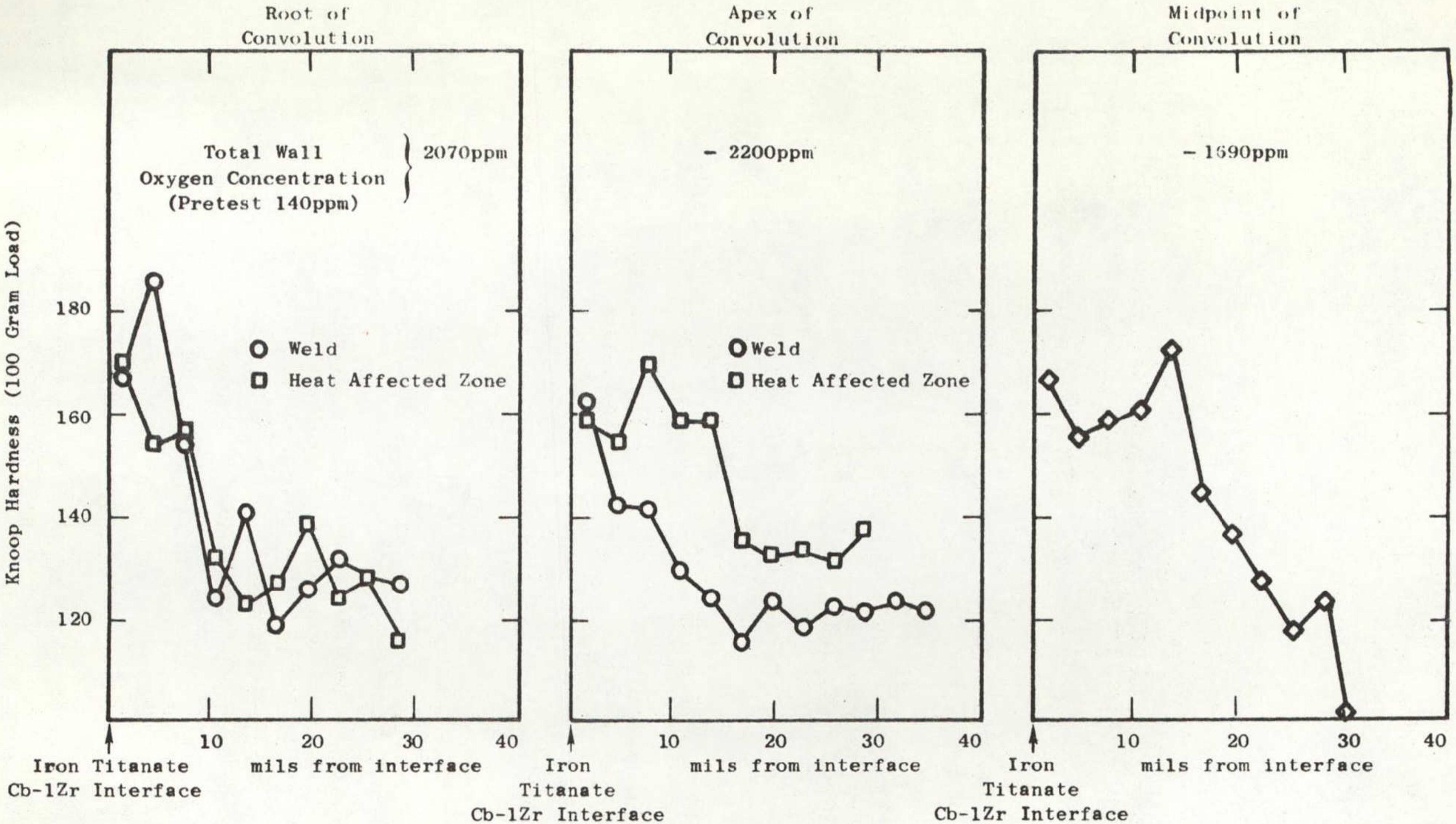


Figure 58. Hardness Surveys of Lithium Fluoride Bellows Capsule II After 3000 Hours of Testing. Pretest Knoop Hardness of the Cb-1Zr was 80.

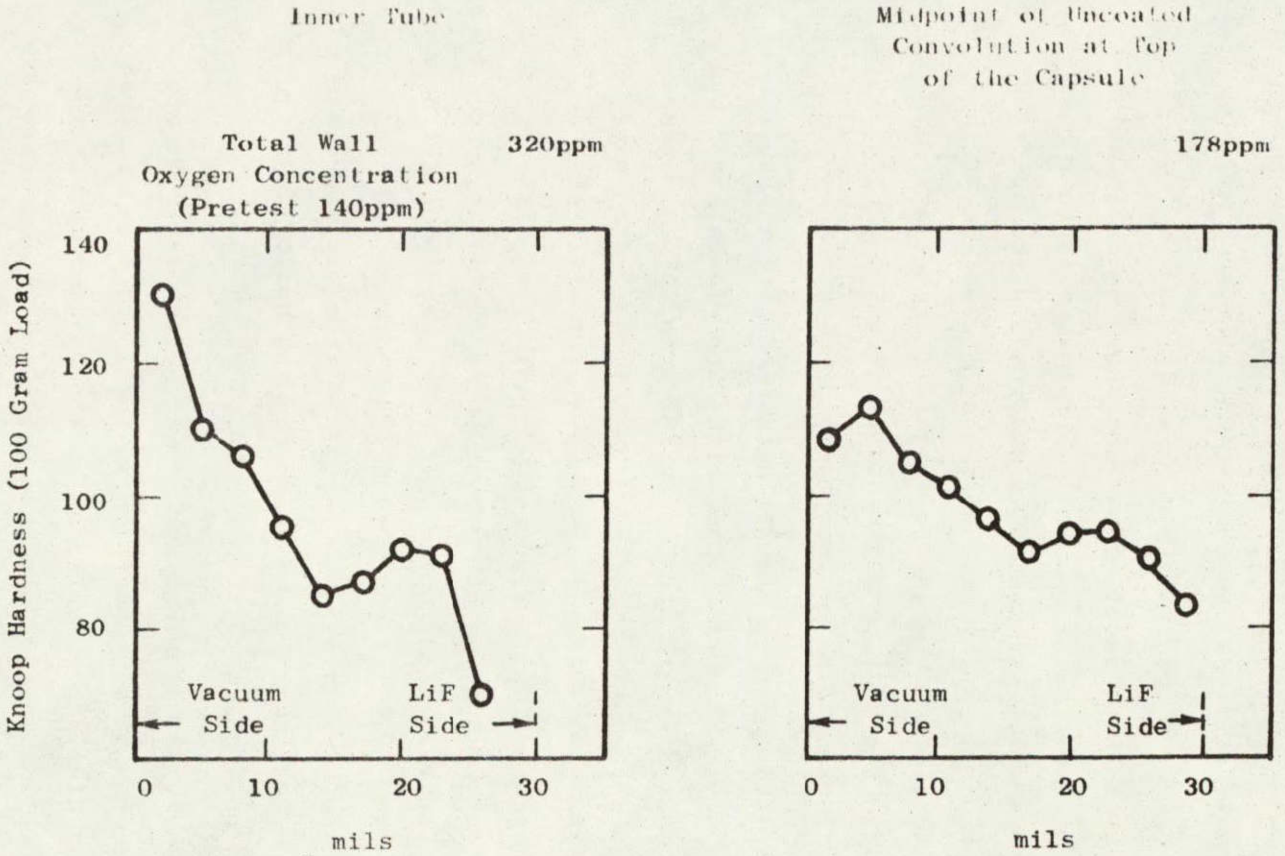


Figure 59. Hardness Surveys of Lithium Fluoride Bellows Capsule II. After 3000 Hours of Testing. Pretest Knoop Hardness of the Cb-1Zr was 80.

hardness values observed near the tube surface are attributed to oxygen contamination from the iron titanate coating although the surface was not in contact with the coating on the stainless steel tube.

Microprobe analysis of specimens removed from the walls of Bellows Capsule II was performed at GE with an Applied Research Laboratory EMX Electron Microprobe X-ray Analyzer. X-ray wavelength scans of the iron titanate coating were made on both pretest and posttest specimens. The following elements were identified; iron, titanium, aluminum, silicon and manganese.

Electron back scatter (EBS) oscillograms are shown which describe the overall image of the areas being scanned. The images in this case are produced by electrons which have not been absorbed in the specimen. The absorption of electrons is a function of the atomic weight of the elements bombarded. The greater the atomic weight of the elements which are present in the microstructural constituents the lighter the resulting image on the positive electron back scatter oscillogram.

Oscillograms were made for each element detected by the x-ray wavelength scans and are shown in Figures 60 and 61. Each element scan was performed on the identical area illustrated in EBS scan. The images are produced by the characteristic x-rays of that element which have been produced by electron bombardment. A comparison of the before test and after test oscillograms shown in Figure 60 reveals some distinct changes in the iron titanate coating after 3000 hours of testing. Before testing iron and titanium were distributed uniformly throughout the coating as iron titanate. As result of thermal cycling between 1500 and 1700°F the coating has substantially decomposed and iron has segregated into nodules within the coating. The titanium remains uniformly dispersed throughout the coating around the iron nodules.

Oscillograms for silicon, aluminum and manganese, shown in Figure 61, indicate these elements to be impurities in the iron titanate. The higher concentration of aluminum and silicon at the coating-substrate interface most likely is a result of aluminum oxide and silicon carbide particles imbedded in the Cb-1Zr surface during the grit blasting which

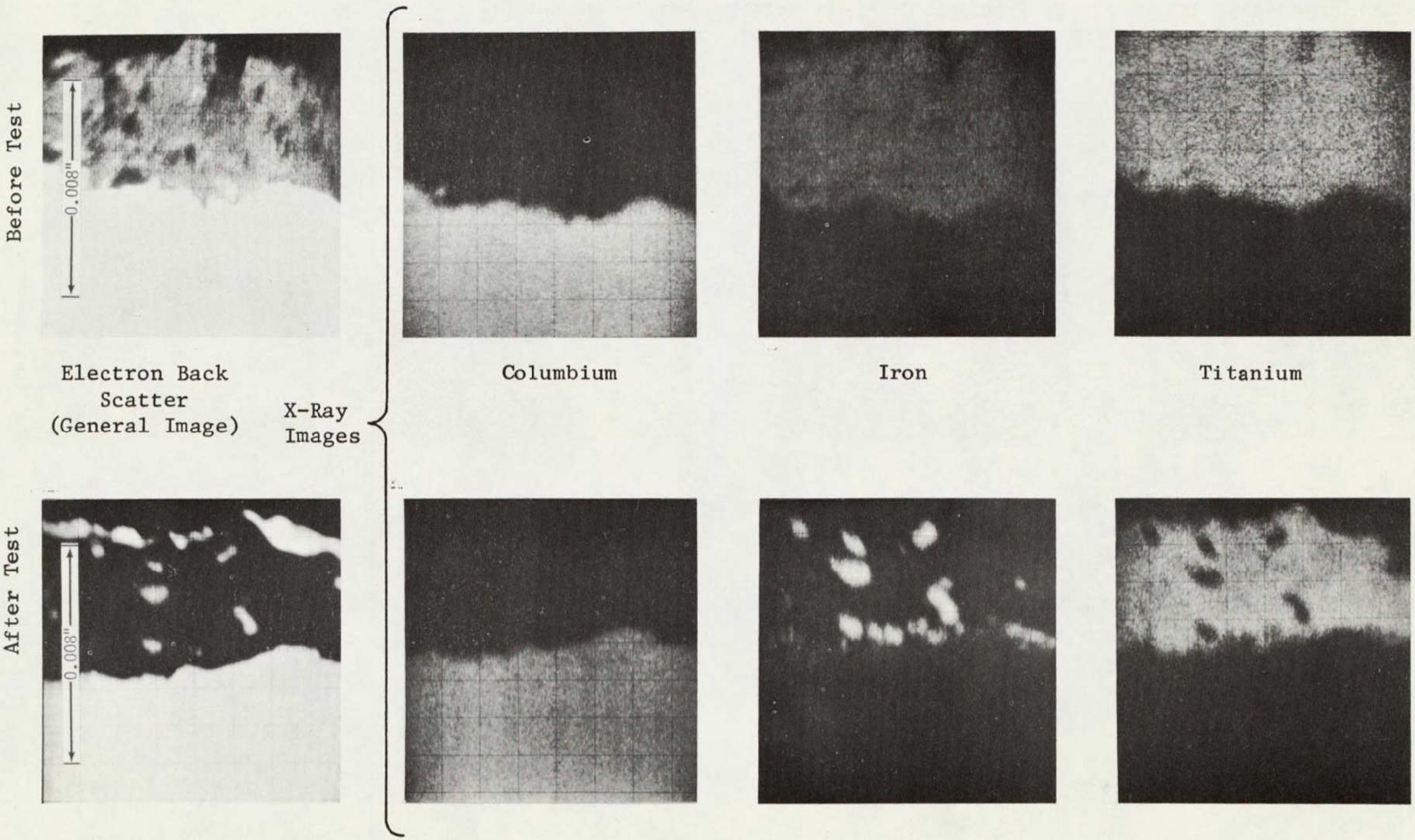


Figure 60. Electron Microprobe Oscillograms Showing the Distribution of Elements in Iron Titanate Coated Cb-1Zr. Specimens were Removed from the Wall of Bellows Capsule II After 3000 Hours of Testing - 1875 Thermal Cycles Between 1500 and 1700°F.

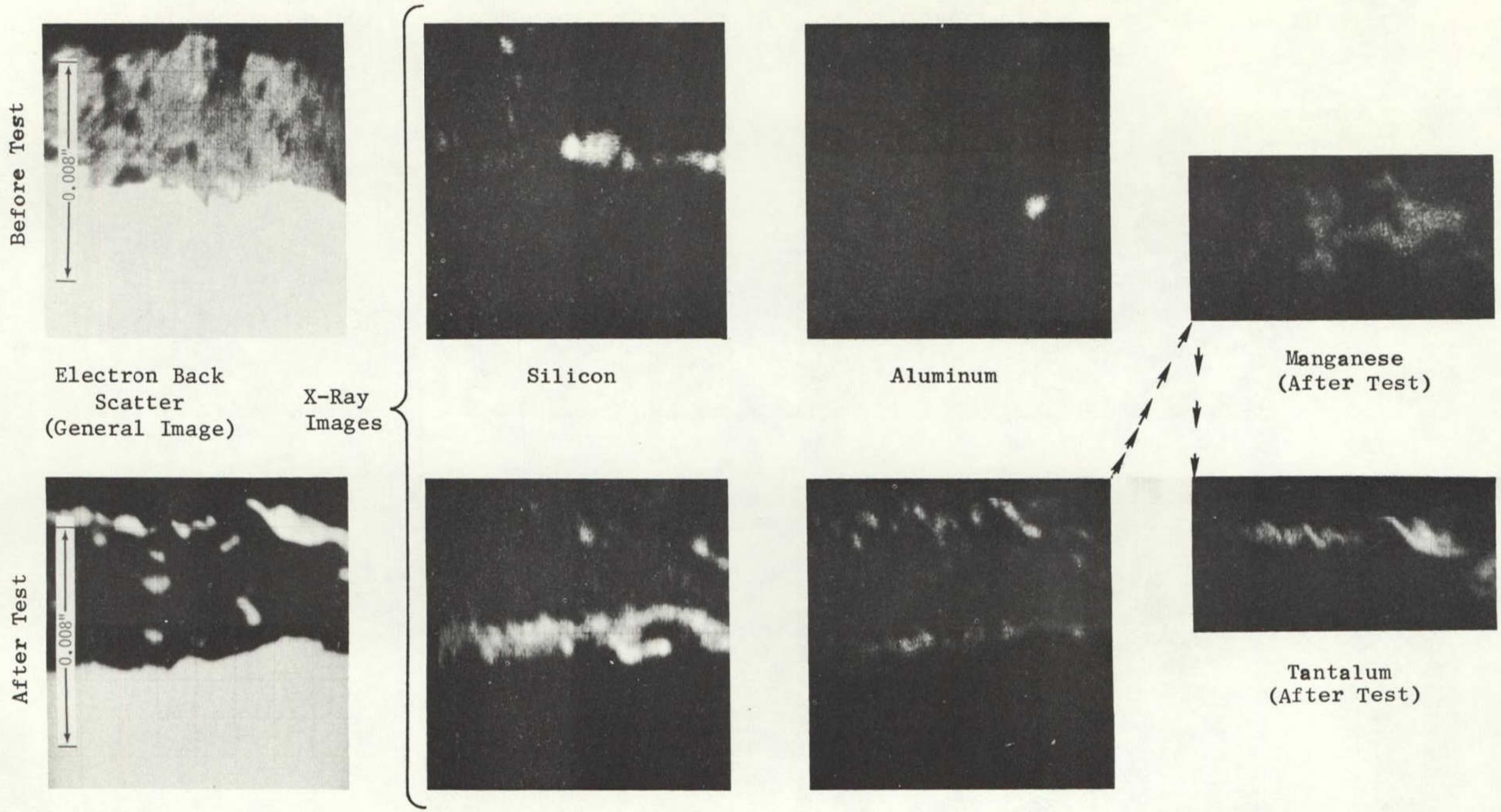


Figure 61. Electron Microprobe Oscillograms Showing the Distribution of Elements in Iron Titanate Coated Cb-1Zr. Specimens were Removed from the Wall of Bellows Capsule II After 3000 Hours of Testing - 1875 Thermal Cycles Between 1500 and 1700°F.

preceded the application of the coating. A slight amount of tantalum was observed at the surface of coating after testing. This tantalum surface deposit is attributed to vapor deposition from the split tantalum heating element which operated at a temperature as high as 3500°F.

As described previously the grain boundary phase noted in the posttest Cb-1Zr microstructure just below the iron titanate coating was tentatively identified as columbium nitride by stain etching techniques. Microprobe traverses of this phase, shown in the photomicrographs in Figure 57, indicated columbium as the only detectable element. The sensitivity of the microprobe for nitrogen is limited (> 1000 ppm).

C. BELLOWS CAPSULE III

The posttest evaluation of Bellows Capsule III included chemical analysis, metallography, x-ray diffraction and microprobe. The capsule was sectioned and lithium fluoride was removed in the same manner as Bellows Capsule II. The sectioned capsule is shown in Figure 62. The areas selected for posttest evaluation are shown in Figure 63.

1. Chemical Analysis

Two set of specimens from the 5th and 2nd convolutions were analyzed for oxygen, nitrogen, and hydrogen. One set of specimens was analyzed with the grit blasted surface intact - while the other set of specimens was analyzed after the grit blasted surface had been removed by filing. The results, shown in Table XV, indicate the specimens with the grit blasted surface left intact showed considerably higher oxygen concentration than those that had the grit blasted surface removed. The higher oxygen analysis is attributed to the alumina grit particles imbedded in the surface. Pretest specimens that had been grit blasted showed the same high oxygen concentration. The analytical results indicate oxygen contamination by the alumina grit was limited to the surface of the convolutions.

2. Metallography

Specimens for metallographic examination were removed from the 1st and 5th convolutions, which represent the coolest and hottest areas of the capsule. Examination of these specimens showed no evidence of

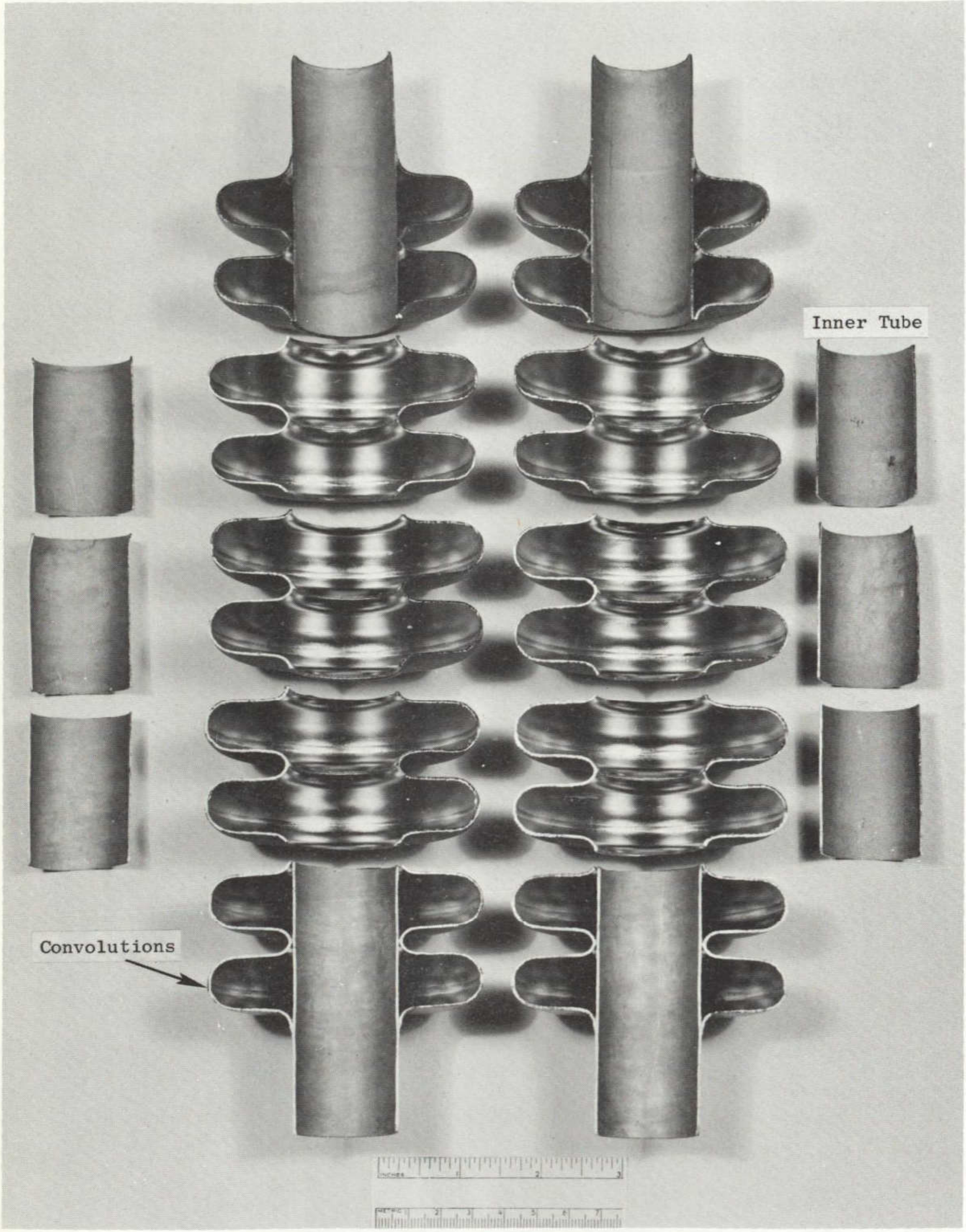


Figure 62. Lithium Fluoride Bellows Capsule III Sectioned After 5000 Hours of Testing. The Residual Lithium Fluoride was Removed from the Cb-1Zr Capsule with Hot Water. (69-10-25B)

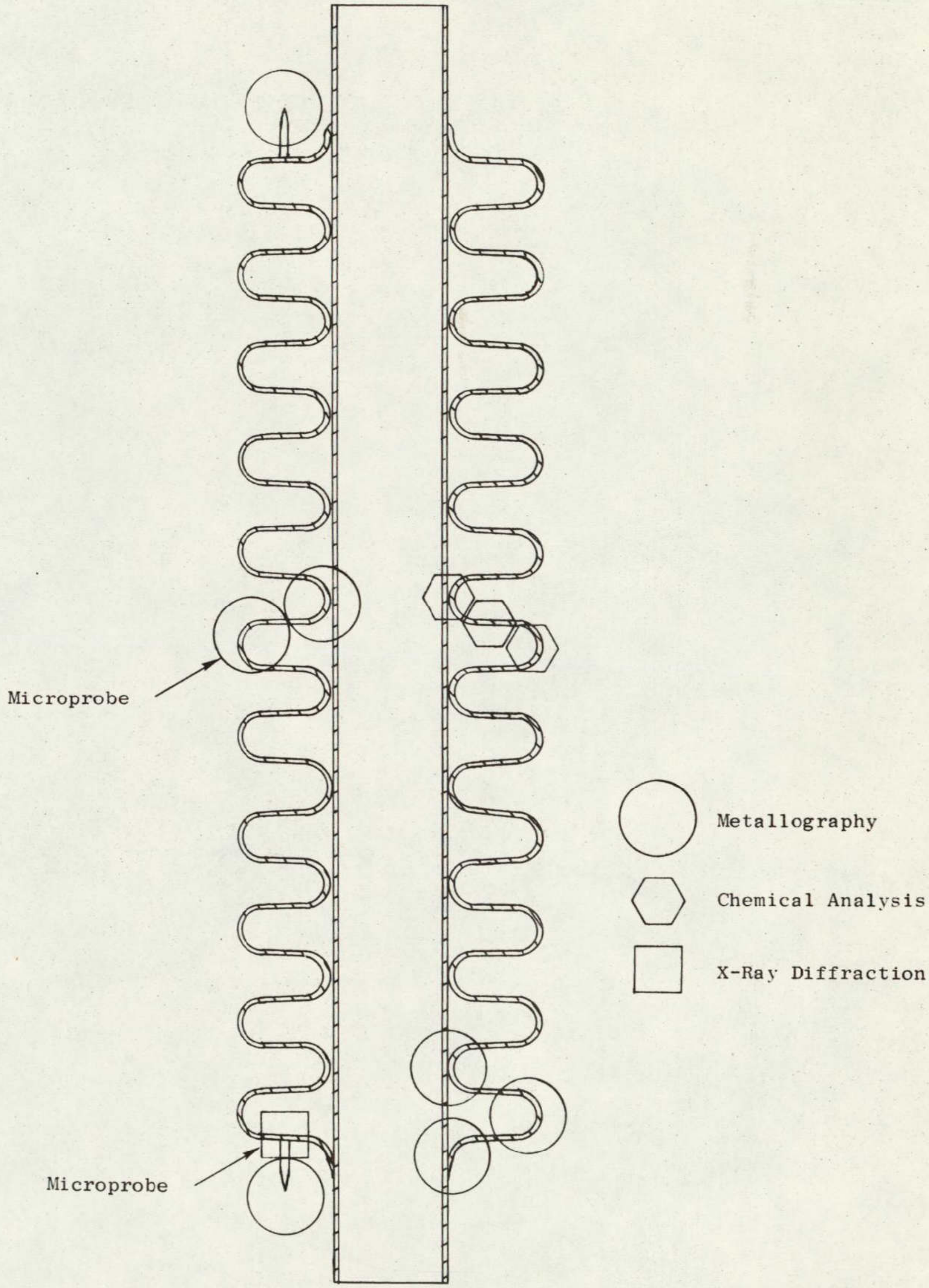


Figure 63. Location of Posttest Evaluation Specimens Taken from Bellows Capsule III.

TABLE XV.

INTERSTITIAL ANALYSIS OF Cb-1Zr CONVOLUTIONS REMOVED FROM BELLOWS CAPSULE III

		Concentration, ppm					
		Pretest		After 5000 Hours of Testing			
				Convolution #2		Convolution #5	
Area of Convolution		Before Grit Blasting	As Grit Blasted	As Grit Blasted	Grit Blasted Surface Removed	As Grit Blasted	Grit Blasted Surface Removed
Apex	O	174 124 (149)[a]	270 259 (265)	314 323 (319)	137	164 160 (162)	101 102 (102)
	N	25 15 (20)	64 58 (61)	24 18 (21)	27	21 16 (19)	44 29 (37)
	H	5 2	11 7	1 6	< 1	< 1 < 1	< 1
Midpoint	O	140 151 (146)	250 244 (247)	226 158 (192)	139	96 130 (113)	109 93 (101)
	N	23 50 (37)	42 51 (47)	25 13 (19)	19	18 17 (18)	41 26 (34)
	H	1 4	3 7	< 1 < 1	< 1	< 1 < 1	< 1
Root	O		363 344 (353)	189 190 (190)	100	232 192 (212)	104
	N		40 48 (44)	20 20 (20)	13	27 24 (26)	45
	H		6 4	< 1 < 1	< 1	2 1	< 1

[a]() average

either corrosion or contamination as can be seen from comparison of pretest and posttest specimens shown in Figures 64 and 65. While no serious effects of aluminum oxide grit blasting was noted, a slight reaction between the alumina particles imbedded in the Cb-1Zr did occur. This reaction zone is shown in the as-polished microstructure in Figure 66. The same area after etching is also shown, but the reaction zone was attacked and removed by the etchant.

After sectioning Bellows Capsule III and removing the lithium fluoride, a satiny-gray colored area (shown in Figure 67) was noted in and around one fill tube. Photomicrographs of a cross section of this fill tube are shown in Figure 68, and indicate a thin continuous phase on the ID of the fill tube. This phase was not found on the other fill tube. When stain etching techniques were applied to identify this phase the color produced indicated both an oxide and nitride.[6]

3. X-Ray Diffraction and Microprobe Analyses

The phase observed on the ID of the fill tube (Figure 67) was analyzed by x-ray diffraction and electron microprobe techniques. X-ray diffraction analysis indicated the phase to be columbium nitride oxide which has a face centered cubic lattice with a lattice parameter 4.42 angstroms.[7] Microprobe analysis scans across this phase indicated the same ratio of columbium and zirconium as found in the matrix. Identification of oxygen and nitrogen was not attempted because of the microprobe limited sensitivity for the light elements (> 1000 ppm). The phase is believed to be (Cb,Zr)-O-N, and it is postulated that it was produced as a result of contamination during filling of the capsule. Since the phase was only found around one fill tube the contamination could have resulted from the partial pressure of oxygen and nitrogen inside the capsule being concentrated at the top fill tube as lithium fluoride was emitted from the bottom of the capsule. The capsule temperature during filling was 1775°F, sufficiently high to cause the oxide nitride to form if sufficient oxygen and nitrogen were present.

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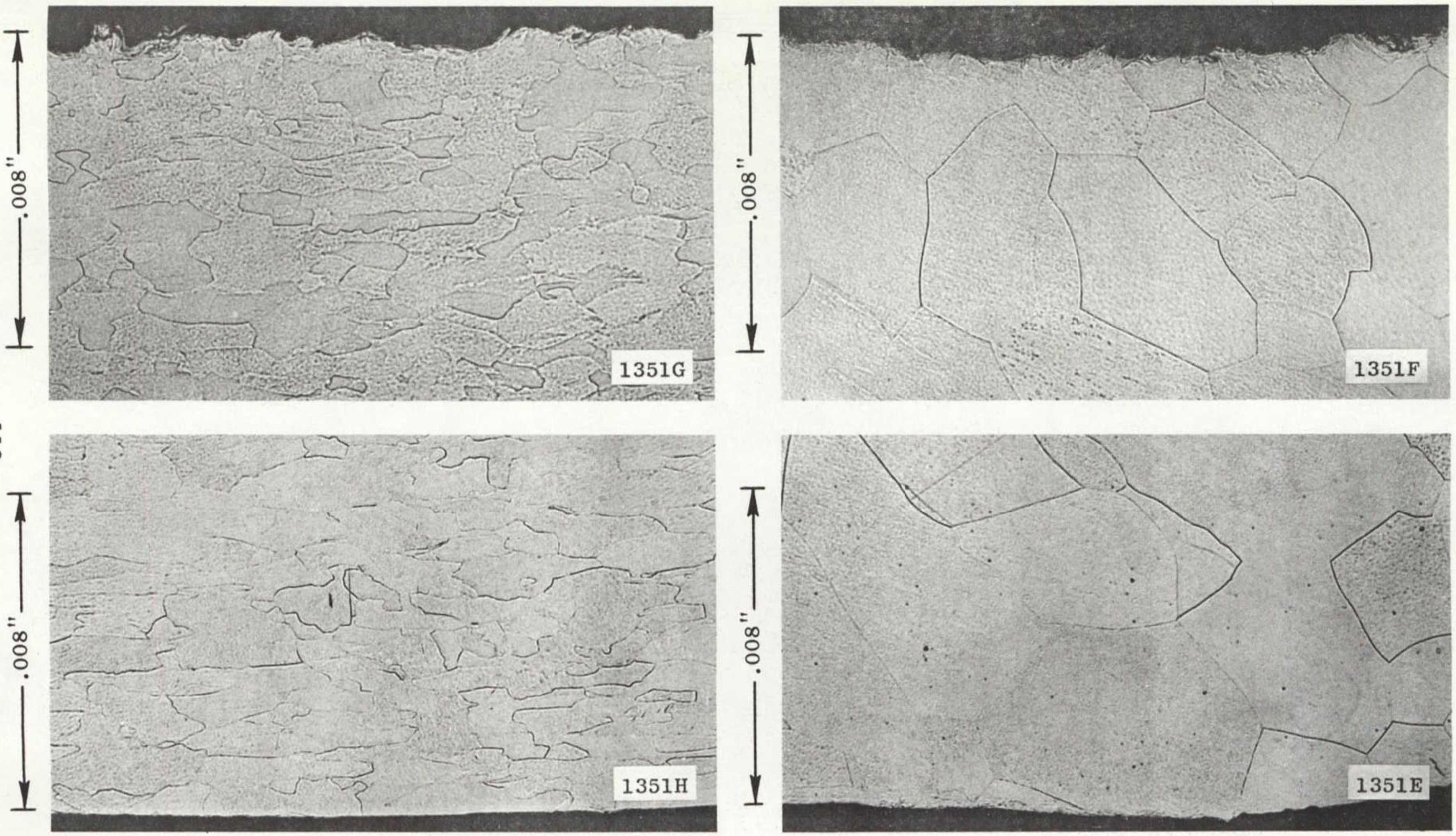
[6] Crouse, R. S., "Identification of Carbides, Nitrides and Oxides of Niobium and Niobium Alloys by Anodic Staining," ORNL-3821, July 1965.

[7] Schoenberg, Acta Chem, Scan. 8 208 (1954)

Gritblasted Surface

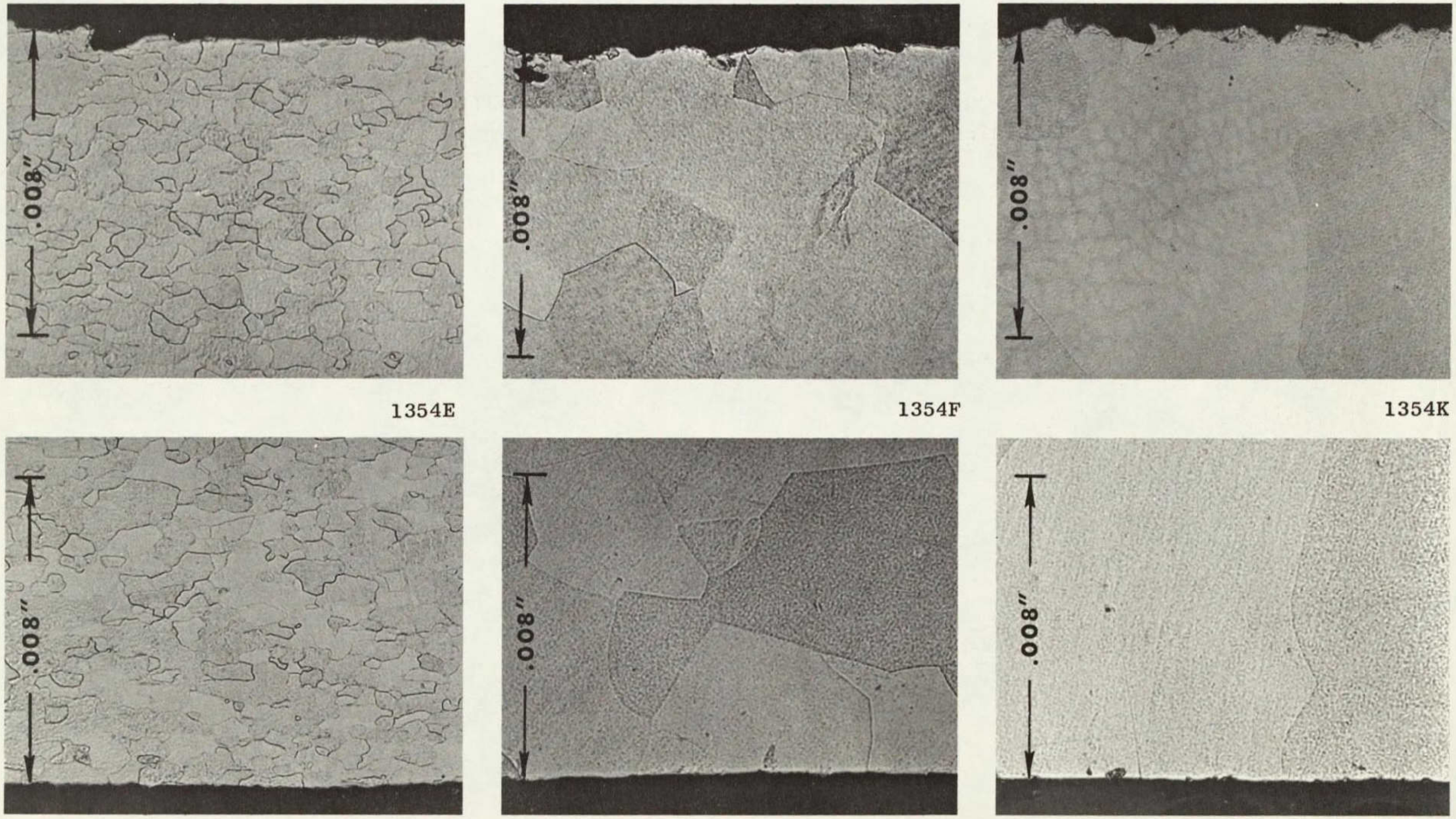


Parent Metal

Weld

Figure 64. Photomicrographs of Pretest Cb-1Zr Specimen After Gritblasting with Al_2O_3 in the Same Manner as the Bellows Capsule III. Etchant: 60% Glycerin, 20% HNO_3, 20% HF.

Vacuum Side (Gritblasted Surface)



1354E 1354F 1354K

Parent Metal 1354D

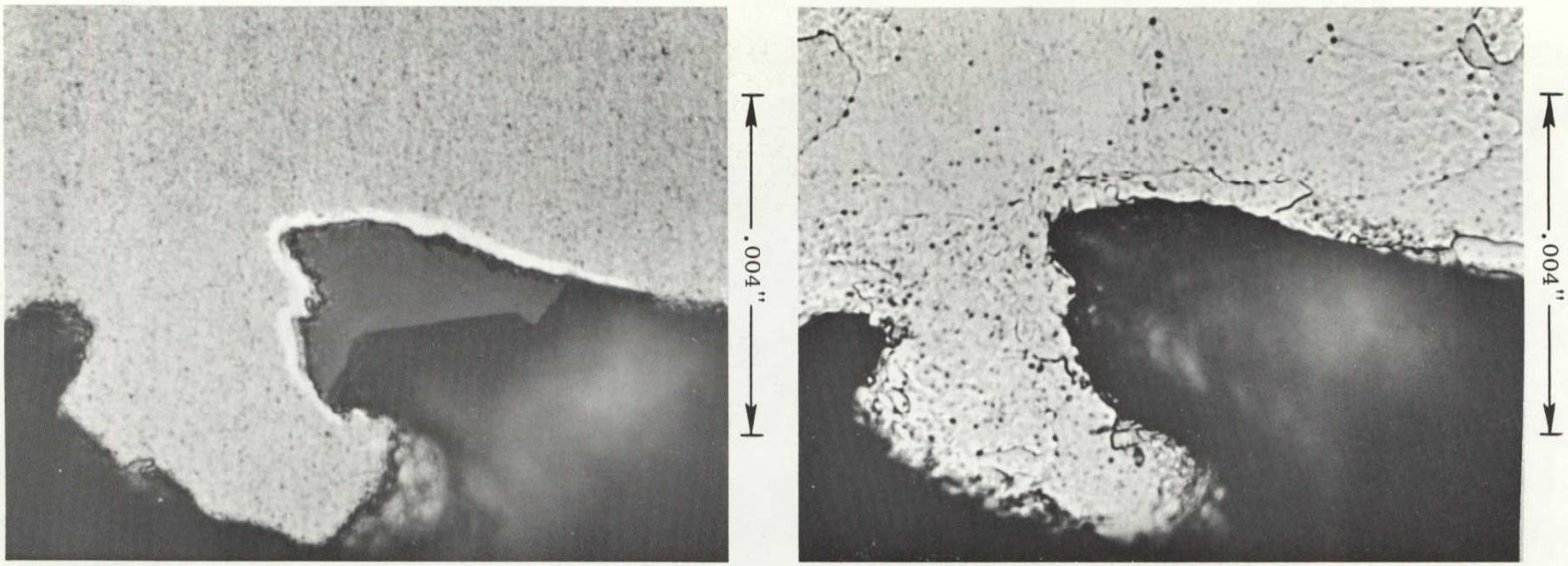
Heat Affected Zone 1354J

Weld 1354L

Lithium Fluoride Side

Figure 65. Photomicrographs of 5th Convolution From Cb-1Zr Bellows Capsule III After 5000 Hours of Testing. Maximum Lithium Fluoride Temperature 1700°F.

Etchant: 60% Glycerine, 20% HNO_3, 20% HF



As Polished 1354B

Etched 1354H

Figure 66. The Surface of a Cb-1Zr Convolution on the Vacuum Side Following 5000 Hours of Testing. The Reaction Zone Around the Alumina Particle, Imbedded in the Surface During Grit-Blasting, was Identified as a Columbium Oxide. Etchant: 60% Glycerine, 20% HNO_3, 20% HF

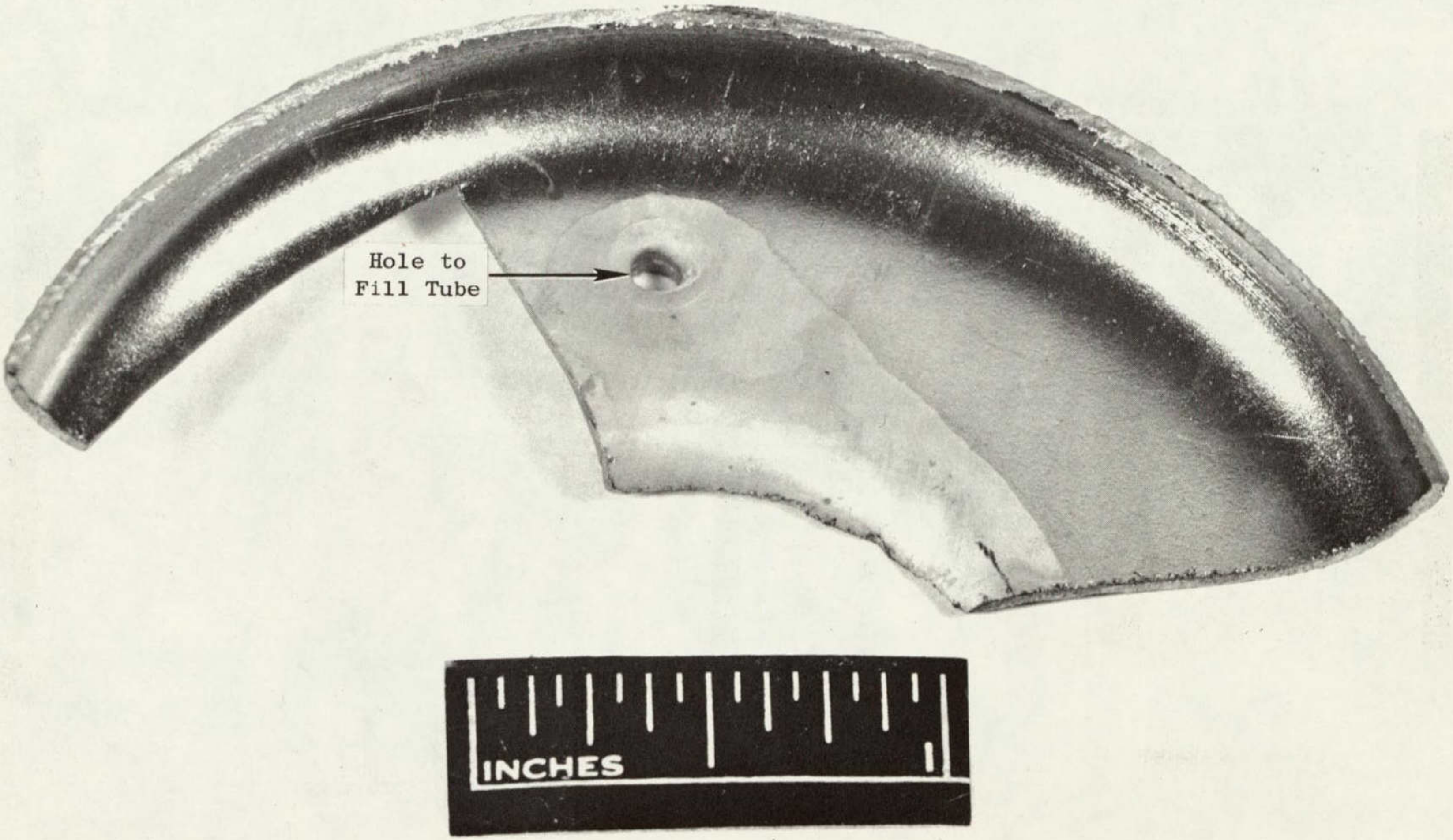


Figure 67. Contaminated Area Around Upper Fill Tube and Top Convolution of Bellows Capsule III. The Surface Phase was Identified as a Cb-O-N Compound. (P69-12-7)

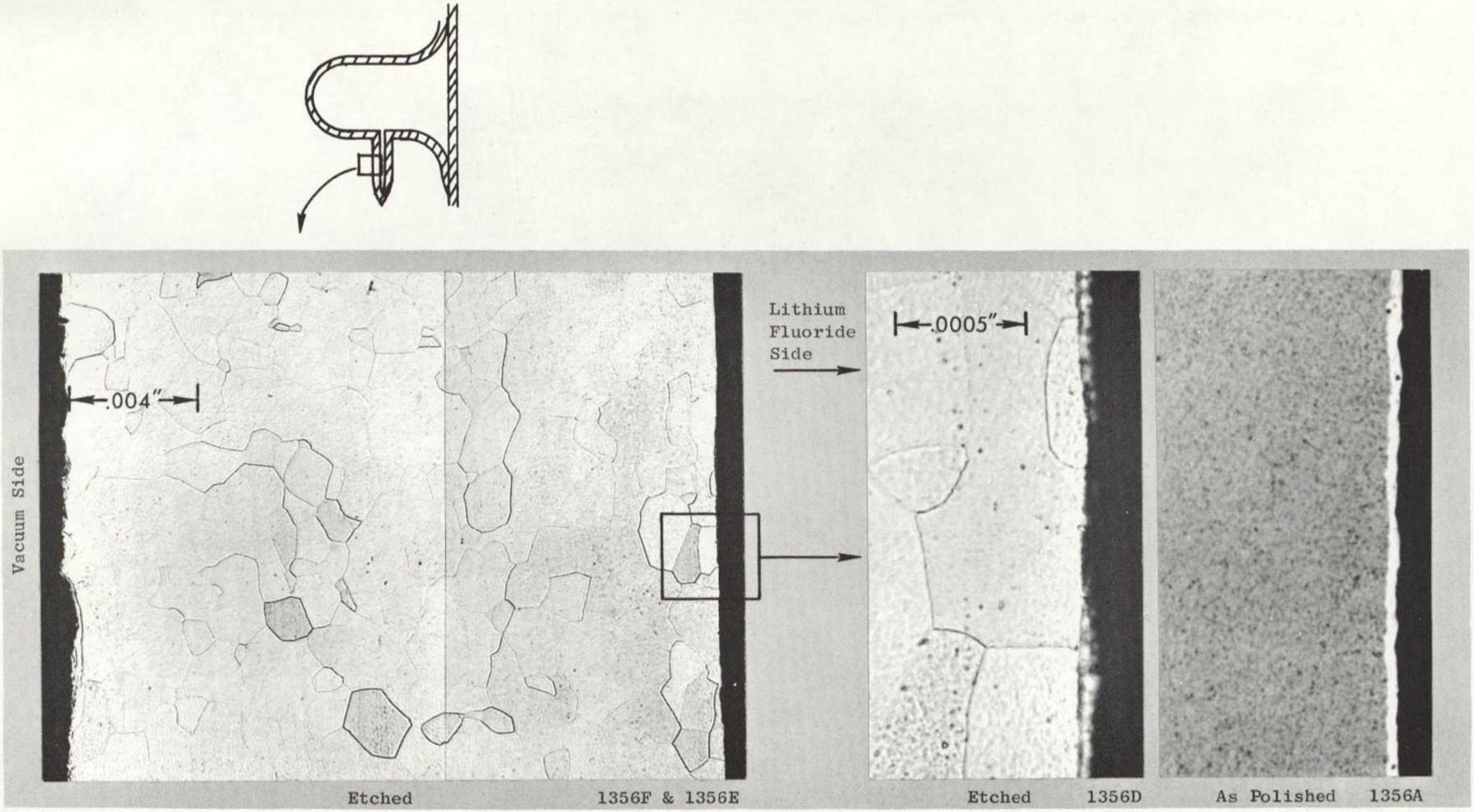


Figure 68. Photomicrographs of Fill Tube From Bellows Capsule III After 5000 Hours Test Time.

Microprobe analysis of the grit blasting particle embedded in the outside surface of a Cb-1Zr convolution (Figure 66) identified the material as alumina. The reaction zone below the particle contained columbium and zirconium in the same ratio as the matrix and is believed to be (Cb, Zr) O.

IV. SUMMARY AND CONCLUSIONS

The following three Cb-1Zr bellows capsules containing lithium fluoride were tested:

Bellows Capsule I - was cycled between 1500° and 1900°F for 1000 hours.

Bellows Capsule II - was coated with iron titanate and cycled between 1500° and 1700°F for 3000 hours.

Bellows Capsule III - was grit blasted with alumina and cycled between 1500° and 1700°F for 5000 hours.

The posttest evaluation of these capsules which included metallography, chemistry, microprobe analysis, x-ray diffraction, and dimensional analysis revealed the following:

1. The bellows design does accomplish the purpose of compartmentalizing the lithium fluoride and provide a void volume to accommodate the 29.4% increase in volume of lithium fluoride that occurs on melting.
2. Thermal cycles between 1500°F and 1900°F for 1000 hours caused severe distortion of the Cb-1Zr bellows capsule, while capsules cycled between 1500°F and 1700°F for times up to 5000 hours showed only minor deformation.
3. The iron titanate coating on Bellows Capsule II showed serious deterioration and contaminated the Cb-1Zr substrate. Use of iron titanate was eliminated from consideration for the heat receiver.
4. Aluminum oxide grit blasting of the Cb-1Zr had no serious metallurgical effects on the Cb-1Zr capsule material and was selected for use on the heat receiver.

In conclusion, Cb-1Zr heat receiver tubes containing lithium fluoride can be expected to give satisfactory service when thermal cycled between 1500°F and 1700°F for times up to 5000 hours.

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